

**SAF-RC-232**  
**100-IU-2 & 100-IU-6 Remaining**  
**Waste Sites – Soil Full Protocol**  
**FINAL VALIDATION PACKAGE**

**COMPLETE COPY OF VALIDATION PACKAGE TO:**

No Distribution Required

KW 10/27/15  
INITIAL/DATE

**COMMENTS:**

**SDG JP1000**

**SAF-RC-232**

**Sample Location: 600-326**

Date: 19 August 2015  
To: Washington Closure Hanford Inc. (technical representative)  
From: ELR Consulting  
Project: 100-IU-2 & 100-IU-6 Remaining Waste Sites – Soil Full Protocol - Waste Site 600-326  
Subject: Inorganic - Data Package No. JP1000-TAL

## **INTRODUCTION**

This memo presents the results of data validation on Data Package No. JP1000 prepared by TestAmerica Laboratories (TAL). A list of samples validated along with the analyses reported and the method of analysis is provided in the following table.

Sample ID	Sample Date	Media	Validation	Analyte
J1V846	10/1/15	Soil	C	See note 1
J1V847	10/1/15	Soil	C	See note 1
J1V848	10/1/15	Soil	C	See note 1
J1V849	10/1/15	Soil	C	See note 1

1 - ICP metals (6010B).

Data validation was conducted in accordance with the Washington Closure Hanford (WCH) validation statement of work and the 100 Area Remedial Action Sampling and Analysis Plan (DOE/RL-96-22, September 2009). Appendices 1 through 6 provide the following information as indicated below:

- Appendix 1. Glossary of Data Reporting Qualifiers
- Appendix 2. Summary of Data Qualification
- Appendix 3. Annotated Laboratory Reports
- Appendix 4. Laboratory Narrative and Chain-of-Custody Documentation
- Appendix 5. Data Validation Supporting Documentation
- Appendix 6. Additional Documentation Requested by Client

## **DATA QUALITY PARAMETERS**

### **Holding Times**

Analytical holding times for metals are assessed to ascertain whether the holding time requirements were met by the laboratory. The holding time requirements are as follows: Soil samples must be analyzed within 6 months for ICP metals and 28 days for mercury.

All holding times were acceptable.

## · **Preparation (Method) Blanks**

### Preparation Blanks

At least one preparation blank, consisting of deionized distilled water processed through each sample preparation and analysis procedure, must be prepared and analyzed with every sample delivery group. In the case of positive blank results, samples with digestate concentrations less than five times the preparation blank value have had their associated values qualified as non-detected and flagged "UJ". Samples with concentrations of greater than five times the highest blank concentration do not require qualification.

In the case of negative blank results, if the absolute value exceeds the contract required detection limit (CRDL), all nondetects are rejected and flagged "UR" and all detects that are less than ten times the absolute value of the associated preparation blank result are qualified as estimates and flagged "J". If the absolute value of the negative preparation blank is greater than the instrument detection limit (IDL) and less than or equal to the CRDL, all nondetects are qualified as estimates and flagged "UJ" and all detects less than ten times the absolute value of the blank are qualified as estimates and flagged "J". If the sample results are greater than ten times the absolute value of the preparation blank, no qualification is necessary.

Due to method blank contamination, the calcium, chromium, magnesium, zinc and nickel results in sample J1V849 were qualified as undetected and flagged "UJ".

All other preparation blank results were acceptable.

### Field (Equipment) Blank

One field blank (J1V849) was submitted for analysis. Nine analytes were detected in the field blank. Under the WCH statement of work, no qualification is required.

## · **Accuracy**

### Matrix Spike and Laboratory Control Sample

Matrix spike (MS) and laboratory control sample (LCS) analyses are used to assess the analytical accuracy of the reported data. The matrix spike is used to assess the effect of the matrix on the ability to accurately quantify sample concentrations. Recoveries must fall within the range of 75% to 125%. Samples with a recovery of less than 30% and a sample result below the IDL are rejected and flagged "UR". Samples with a recovery of 30% to 74% and a sample result less than the IDL are qualified "UJ". Samples with a recovery of greater than 125% or less than 74% and a sample result greater than the IDL are qualified as estimates and flagged "J". Finally, for samples with a recovery greater than 125% and a sample result less than the IDL, no qualification is required.

Due to matrix spike recoveries outside QC limits, all antimony (52%) and silicon (16%) results were qualified as estimates and flagged "J".

Due to an LCS recovery outside QC limits, all silicon (19%) results were qualified as estimates and flagged "J".

All other accuracy results were acceptable

- **Precision**

Laboratory Duplicate Samples

Analytical precision is expressed by the relative percent differences (RPD) between the recoveries of matrix spike duplicate (MSD) analyses performed on a sample in the analytical batch. Precision may alternatively be assessed using unspiked duplicate analyses performed on a sample in the analytical batch. If both sample and replicate activities (concentrations) are greater than five times the CRDL and the RPD is less than 30%, no qualification is required. If either activity (concentration) is less than five times the CRDL, the RPD control limit is less than or equal to two times the CRDL. If the RPD is outside the applicable control limit, associated results are qualified as estimated detects or estimated non-detects.

All laboratory duplicate results were acceptable.

Field Duplicate

One set of field duplicates (J1V846/J1V848) were submitted for analysis. Field duplicates are compared using the same criteria as for laboratory duplicates. All field duplicate results were acceptable.

- **Analytical Detection Levels**

Reported analytical detection levels are compared against the 100 Area RQLs to ensure that laboratory detection levels meet the required criteria. All results met the RQL.

- **Completeness**

Data package No. JP1000 was submitted for validation and verified for completeness. Completeness is based on the percentage of data determined to be valid (i.e., not rejected). The completion percentage was 100%.

**MAJOR DEFICIENCIES**

None found.

## **MINOR DEFICIENCIES**

The following minor deficiencies were noted:

- Due to method blank contamination, the calcium, chromium, nickel, zinc and magnesium results in sample J1V849 were qualified as undetected and flagged "UJ".
- Due to matrix spike recoveries outside QC limits, all antimony (52%) and silicon (16%) results were qualified as estimates and flagged "J".
- Due to an LCS recovery outside QC limits, all silicon (19%) results were qualified as estimates and flagged "J".

Data flagged "J" indicates that the associated concentration is an estimate, but under the WCH statement of work, the data may be usable for decision-making purposes. All other validated results are considered accurate within the standard error associated with the methods.

## **REFERENCES**

Washington Closure Hanford Contract #S00W307A00 (March 2008), *Data Validation Services*, March 2008.

DOE/RL-96-22, Rev. 5, *100 Area Remedial Action Sampling and Analysis Plan*, U.S. Department of Energy, September 2009.

**Appendix 1**  
**Glossary of Data Reporting Qualifiers**

Qualifiers which may be applied by data validators in compliance with WCH validation SOW are as follows:

- U - Indicates the compound or analyte was analyzed for and not detected in the sample. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory.
- UJ - Indicates the compound or analyte was analyzed for and not detected in the sample. Due to a minor QC deficiency identified during the data validation, the associated quantitation limit is an estimate.
- J - Indicates the compound or analyte was analyzed for and detected. Due to a minor QC deficiency identified during the data validation, the associated concentration is an estimate, but the data are usable for decision-making purposes.
- BJ - Applied to inorganic analyses only. Indicates the analyte concentration was greater than the IDL but less than the CRDL and is considered an estimated value.
- R - Indicates the compound or analyte was analyzed for, detected, and due to an identified major QC deficiency, the data are unusable.
- UR - Indicates the compound or analyte was analyzed for and not detected in the sample. Additionally, the data is unusable due to an identified major QC deficiency.
- NJ - Indicates presumptive evidence of a compound at an estimated value. The data may not be valid for some specific applications (i.e., usable for decision-making purposes).
- N - Indicates presumptive evidence of a compound. The data may not be valid for some specific applications (i.e., usable for decision-making purposes).

**Appendix 2**  
**Summary of Data Qualification**



# INORGANIC DATA QUALIFICATION SUMMARY\*

<b>SDG: JP1000</b>	<b>REVIEWER: ELR</b>	<b>Project: 600-326</b>	<b>PAGE <u>1</u> OF <u>1</u></b>
<b>COMPOUND</b>	<b>QUALIFIER</b>	<b>SAMPLES AFFECTED</b>	<b>REASON</b>
Calcium Chromium Magnesium Nickel Zinc	UJ	J1V849	Method blank contamination
Silicon	J	All	LCS recovery
Antimony Silicon	J	All	MS recovery

\* - The Qualified Data Summary Table includes laboratory applied "U" qualifiers not specifically identified here. The laboratory applied "U" qualifiers are included to minimize misinterpretation of results contained in the table.

**Appendix 3**  
**Annotated Laboratory Reports**

# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Client Sample ID: J1V846

Lab Sample ID: 280-74906-1

Client Matrix: Solid

% Moisture: 9.7

Date Sampled: 10/01/2015 0755

Date Received: 10/02/2015 0935

## 6010B Metals (ICP)

Analysis Method: 6010B

Analysis Batch: 280-298187

Instrument ID: MT\_025

Prep Method: 3050B

Prep Batch: 280-297784

Lab File ID: 25B100615.asc

Dilution: 1.0

Initial Weight/Volume: 1.119 g

Analysis Date: 10/06/2015 1850

Final Weight/Volume: 100 mL

Prep Date: 10/05/2015 1350

*10/12/19*

Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	MDL	RL
Antimony		0.38	U	0.38	0.59
Arsenic		3.5		0.65	0.99
Barium		80.8		0.075	0.49
Beryllium		0.28		0.033	0.20
Boron		1.2	B	0.97	2.0
Cadmium		0.14	B	0.041	0.20
Calcium		3570		13.9	49.5
Chromium		9.4		0.057	0.20
Cobalt		8.1		0.099	0.99
Copper		11.3		0.21	0.99
Lead		6.0		0.27	0.49
Magnesium		4260		3.7	19.8
Manganese		307		0.099	0.99
Molybdenum		0.26	U	0.26	2.0
Nickel		9.8		0.12	4.0
Potassium		1700		40.6	297
Selenium		0.85	U	0.85	0.99
Silicon		325	N	5.6	9.9
Silver		0.16	U	0.16	0.20
Sodium		192		58.4	119
Vanadium		51.9		0.093	2.0

Analysis Method: 6010B

Analysis Batch: 280-298379

Instrument ID: MT\_025

Prep Method: 3050B

Prep Batch: 280-297784

Lab File ID: 25A100715.asc

Dilution: 1.0

Initial Weight/Volume: 1.119 g

Analysis Date: 10/07/2015 1602

Final Weight/Volume: 100 mL

Prep Date: 10/05/2015 1350

Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	MDL	RL
Aluminum		7590		1.5	4.9
Iron		20700	X	3.8	4.9
Zinc		41.8	X	0.39	0.99

# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Client Sample ID: J1V847

Lab Sample ID: 280-74906-2

Client Matrix: Solid

% Moisture: 11.5

Date Sampled: 10/01/2015 0720

Date Received: 10/02/2015 0935

## 6010B Metals (ICP)

Analysis Method: 6010B

Analysis Batch: 280-298187

Instrument ID: MT\_025

Prep Method: 3050B

Prep Batch: 280-297784

Lab File ID: 25B100615.asc

Dilution: 1.0

Analysis Date: 10/06/2015 1900

Initial Weight/Volume: 1.101 g

Prep Date: 10/05/2015 1350

Final Weight/Volume: 100 mL

*M 10/12/15*

Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	MDL	RL
Antimony		0.39	U	0.39	0.62
Arsenic		6.0		0.68	1.0
Barium		113		0.078	0.51
Beryllium		0.32		0.034	0.21
Boron		1.3	B	1.0	2.1
Cadmium		0.35		0.042	0.21
Calcium		5750		14.5	51.3
Chromium		19.1		0.060	0.21
Cobalt		7.8		0.10	1.0
Copper		20.2		0.22	1.0
Lead		12.0		0.28	0.51
Magnesium		5260		3.8	20.5
Manganese		321		0.10	1.0
Molybdenum		0.27	U	0.27	2.1
Nickel		15.5		0.13	4.1
Potassium		1770		42.1	308
Selenium		0.88	U	0.88	1.0
Silicon		328	J	5.8	10.3
Silver		0.16	U	0.16	0.21
Sodium		410		60.6	123
Vanadium		46.9		0.096	2.1

Analysis Method: 6010B

Analysis Batch: 280-298379

Instrument ID: MT\_025

Prep Method: 3050B

Prep Batch: 280-297784

Lab File ID: 25A100715.asc

Dilution: 1.0

Analysis Date: 10/07/2015 1612

Initial Weight/Volume: 1.101 g

Prep Date: 10/05/2015 1350

Final Weight/Volume: 100 mL

Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	MDL	RL
Aluminum		10600		1.6	5.1
Iron		21600	X	3.9	5.1
Zinc		74.6	X	0.41	1.0

## Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Client Sample ID: J1V848

Lab Sample ID: 280-74906-3

Date Sampled: 10/01/2015 0755

Client Matrix: Solid

% Moisture: 8.5

Date Received: 10/02/2015 0935

### 6010B Metals (ICP)

Analysis Method: 6010B

Analysis Batch: 280-298187

Instrument ID: MT\_025

Prep Method: 3050B

Prep Batch: 280-297784

Lab File ID: 25B100615.asc

Dilution: 1.0

Initial Weight/Volume: 1.055 g

Analysis Date: 10/06/2015 1903

Final Weight/Volume: 100 mL

Prep Date: 10/05/2015 1350

*W 10/17/15*

Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	MDL	RL
Antimony		0.39	U	0.39	0.62
Arsenic		3.4		0.68	1.0
Barium		81.8		0.079	0.52
Beryllium		0.28		0.034	0.21
Boron		1.3	B	1.0	2.1
Cadmium		0.10	B	0.042	0.21
Calcium		3570		14.6	51.8
Chromium		8.9		0.060	0.21
Cobalt		9.2		0.10	1.0
Copper		10.8		0.22	1.0
Lead		5.3		0.28	0.52
Magnesium		4160		3.8	20.7
Manganese		154		0.10	1.0
Molybdenum		0.27	U	0.27	2.1
Nickel		9.5		0.13	4.1
Potassium		1720		42.5	311
Selenium		0.89	U	0.89	1.0
Silicon		338	J	5.9	10.4
Silver		0.17	U	0.17	0.21
Sodium		194		61.1	124
Vanadium		49.7		0.097	2.1

Analysis Method: 6010B

Analysis Batch: 280-298379

Instrument ID: MT\_025

Prep Method: 3050B

Prep Batch: 280-297784

Lab File ID: 25A100715.asc

Dilution: 1.0

Initial Weight/Volume: 1.055 g

Analysis Date: 10/07/2015 1614

Final Weight/Volume: 100 mL

Prep Date: 10/05/2015 1350

Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	MDL	RL
Aluminum		7620		1.6	5.2
Iron		20200	X	3.9	5.2
Zinc		42.1	X	0.41	1.0

# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Client Sample ID: J1V849

Lab Sample ID: 280-74906-4

Client Matrix: Solid

% Moisture: 0.0

Date Sampled: 10/01/2015 0743

Date Received: 10/02/2015 0935

## 6010B Metals (ICP)

Analysis Method: 6010B

Analysis Batch: 280-298187

Instrument ID: MT\_025

Prep Method: 3050B

Prep Batch: 280-297784

Lab File ID: 25B100615.asc

Dilution: 1.0

Initial Weight/Volume: 1.129 g

Analysis Date: 10/06/2015 1905

Final Weight/Volume: 100 mL

Prep Date: 10/05/2015 1350

*10/17/15*

Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	MDL	RL
Antimony		0.34	U <i>JS</i>	0.34	0.53
Arsenic		0.58	U	0.58	0.89
Barium		1.3		0.067	0.44
Beryllium		0.029	U	0.029	0.18
Boron		0.87	U	0.87	1.8
Cadmium		0.036	U	0.036	0.18
Calcium		38.3	B <i>JS</i>	12.5	44.3
Chromium		0.16	B <i>JS</i>	0.051	0.18
Cobalt		0.089	U	0.089	0.89
Copper		0.26	B	0.19	0.89
Lead		0.27	B	0.24	0.44
Magnesium		18.9	C <i>JS</i>	3.3	17.7
Manganese		3.0		0.089	0.89
Molybdenum		0.23	U	0.23	1.8
Nickel		0.17	B C <i>JS</i>	0.11	3.5
Potassium		36.3	U	36.3	266
Selenium		0.76	U	0.76	0.89
Silicon		91.5	<i>JS</i>	5.0	8.9
Silver		0.14	U	0.14	0.18
Sodium		52.3	U	52.3	106
Vanadium		0.25	B	0.083	1.8

Analysis Method: 6010B

Analysis Batch: 280-298379

Instrument ID: MT\_025

Prep Method: 3050B

Prep Batch: 280-297784

Lab File ID: 25A100715.asc

Dilution: 1.0

Initial Weight/Volume: 1.129 g

Analysis Date: 10/07/2015 1617

Final Weight/Volume: 100 mL

Prep Date: 10/05/2015 1350

Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	MDL	RL
Aluminum		112		1.4	4.4
Iron		171	X	3.4	4.4
Zinc		1.3	C X <i>JS</i>	0.35	0.89

## **Appendix 4**

### **Laboratory Narrative and Chain-of-Custody Documentation**

## CASE NARRATIVE

Client: Washington Closure Hanford

Project: WASHINGTON CLOSURE HANFORD

Job Number: 280-74906-1

SDG #: JP1000

SAF#: RC-232

Date SDG Closed: October 2, 2015  
Data Deliverable: 7 Day / Summary

<u>CLIENT ID</u>	<u>LAB ID</u>	<u>ANALYSES REQUESTED</u>	<u>ANALYSES PERFORMED</u>
J1V846	280-74906-1	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V847	280-74906-2	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V848	280-74906-3	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V849	280-74906-4	6010	6010B

I certify that this data package is in compliance with the SOW, both technically and for completeness, for other than the conditions detailed in this Case Narrative. Release of the data contained in this hard copy data package has been authorized by the Laboratory Manager or a designee, as verified by the signature on the Report Cover.

With exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. All laboratory quality control samples analyzed in conjunction with the samples in this project were within established control limits, with any exceptions noted. Calculations are performed before rounding to avoid round-off errors in calculated results.

This report includes reporting limits (RLs) less than TestAmerica Denver's practical quantitation limits. These reporting limits are being used specifically at the client's request to meet the needs of this project. Please note that data are not normally reported to these levels without qualification, since they are inherently less reliable and potentially less defensible than required by the current NELAC standards.

The results, RLs and MDLs included in this report have been adjusted for dry weight, as appropriate.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

### RECEIPT

The samples were received on 10/2/2015 9:35 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperature of the cooler at receipt was 3.3° C.

### GC SEMIVOLATILES - SW846 8081A - Pesticides

The organic prep laboratory noted that the samples presented in this report required a Florisil clean-up to reduce matrix interferences.

The laboratory noted that the samples presented in this report required a mercury clean-up to reduce matrix interferences caused by sulfur.

The RPD between the primary and confirmation columns exceeded 40% for 4,4'-DDE in sample J1V847. The higher of the two values has been reported. The result has been flagged with a "Y".

The MS/MSD performed on sample J1V846 exhibited spike compound recoveries outside the control limits, and the associated sample results have been flagged "N". In addition, the RPD limit was exceeded for 4,4'-DDD and 4,4'-DDE. The acceptable LCS analysis data indicated that the analytical system was operating within control; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

### HPLC - SW846 8310 - PAHs

The RPD between the primary and confirmation columns exceeded 40% for Benzo[a]anthracene and Phenanthrene in sample J1V847. The lower of the two values has been reported, as matrix interference is evident on both columns. The results have been flagged with an "X".



The MS/MSD performed on sample J1V847 exhibited percent recoveries outside the control limits for Chrysene, and the associated sample result has been flagged "N". The acceptable LCS analysis data indicated that the analytical system was operating within control; therefore, corrective action is deemed unnecessary.

Continuing Calibration Verification (CCV) standards associated with samples in analysis batch 280-298048 exhibited %Difference (%D) values >15%, biased high, for Benzo[g,h,i]perylene, Dibenzo[a,h]anthracene and Fluoranthene. The samples associated with these CCVs are either non-detect or less than the reporting limit; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

#### **TOTAL METALS - SW846 6010B**

Serial dilution of a digestate in batch 280-297784 indicates that physical and chemical interferences are present for Iron and Zinc. Results have been flagged with an "X".

Low levels of Barium, Calcium, Chromium, Magnesium and Nickel are present in the method blank associated with batch 280-297784. Because the concentrations in the method blank are not present at levels greater than half the reporting limit, corrective action is deemed unnecessary.

Zinc is present in the method blank associated with batch 280-297784 at 0.797 mg/kg, which is greater than half the project specific reporting limit (PSRL) of 1 mg/kg. TestAmerica's practical quantitation limit (PQL) for Zinc is 3 mg/kg. The laboratory cannot maintain system cleanliness at this low level; therefore, corrective action is not initiated. It can be noted that the concentration found in the method blank is less than half of the laboratory standard PQL, and with the exception of 'blank' sample J1V849, the associated sample amounts are greater than twenty times the method blank concentration.

Iron, a common laboratory contaminant, is present at a level greater than the reporting limit in the method blank associated with batch 280-297784. As the associated sample amounts are greater than twenty times the method blank concentration, corrective action is deemed unnecessary.

It can be noted that the sample amount was greater than four times the spike amount for Aluminum, Iron and Manganese in the Matrix Spike performed on sample J1V846; therefore, control limits are not applicable.

Silicon was recovered outside the control limits in the Matrix Spike performed on sample J1V846, and the associated sample result has been flagged "N". There is no indication that the analytical system was operating out of control, and method accuracy has been verified by the acceptable LCS analysis data; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

#### **GENERAL CHEMISTRY - SW846 9056M - ANIONS**

Each sample is analyzed to achieve the lowest possible reporting limits within the constraints of the method. Due to high constituent concentration, the Sulfate analysis of sample J1V847 had to be performed at a dilution, and the associated result has been flagged with a "D". The reporting limit has been adjusted relative to the dilution required.

Sulfate is present in the method blank associated with batch 280-297863 at 7.21 mg/kg, which is greater than the project specific reporting limit (PSRL) of 5 mg/kg. TestAmerica's practical quantitation limit (PQL) for Sulfate is 50 mg/kg. The laboratory cannot maintain system cleanliness at this low level; therefore, corrective action is not initiated. It can be noted that the concentration found in the method blank is less than half of the laboratory standard PQL.

The Matrix Spike performed on sample J1V846 exhibited the percent recovery outside the control limits for Fluoride, and the associated sample result has been flagged "N". There is no indication that the analytical system was operating out of control, and method accuracy has been verified by the acceptable LCS analysis data; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

Washington Closure Hanford		CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST				RC-232-102		Page 1 of 1	
Collector <b>CRAIG, JC</b>		Company Contact <b>Joan Kessner</b>		Telephone No. <b>375-4688</b>		Project Coordinator <b>KESSNER, JH</b>		Price Code <b>83</b>	
Project Designation <b>100-IU-2 &amp; 100-IU-6 Remaining Waste Sites</b>		Sampling Location <b>600-326 (Areas 1 and 2) verification</b>		SAF No. <b>RC-232</b>		Data Turnaround <b>7 days</b>			
Ice Chest No. <b>AFS-04-008</b>		Field Logbook No. <b>EL-1667-03</b>		COA <b>0603262000</b>		Method of Shipment <b>Commercial Carrier</b>		<b>1 Fed Ex</b>	
Shipped To <b>TestAmerica Denver</b>		Offsite Property No. <b>A131443</b>		Bill of Lading/Air Bill No. <b>See ASPC</b>					
Other Labs Shipped To <b>NIA TRG 10-1-15</b>		Preservation		Cool <=6C	Cool <=6C	Cool <=6C	Cool <=6C		
		Type of Container		G/P	aG	aG	G/P		
POSSIBLE SAMPLE HAZARDS/REMARKS  None		No. of Container(s)		1	1	1	1		
		Volume		250mL	250mL	250mL	250mL		
		Sample Analysis		See Item (1) in Special Instructions	PAHs - 8310	Pesticides - 8061	See Item (2) in Special Instructions		
Special Handling and/or Storage <b>Cooling as required</b>									
Page 1		Sample No.	Matrix	Sample Date	Sample Time				
		J1V846	SOIL	10/01/15	0755	X	X	X	X
		J1V847	SOIL	10/01/15	0720	X	X	X	X
		J1V848	SOIL	10/01/15	0755	X	X	X	X
		J1V849	SOIL	10/01/15	0743	X			
CHAIN OF POSSESSION					SPECIAL INSTRUCTIONS				
Relinquished By/Removed From		Date/Time		Received By/Stored In		Date/Time		<p>(1) ICP Metals - 6010TR (Close-out List) (Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silicon, Silver, Sodium, Vanadium, Zinc)</p> <p>(2) IC Anions - 9056 Modified (Bromide, Chloride, Fluoride, Nitrogen in Nitrate, Nitrogen in Nitrite, Phosphorous in phosphate, Sulfate)</p> <p>3.2 IKS + 0.1</p> <p>T. L. by M7 10-12-15</p>	
Jim C. [Signature]		10/1/15 0808		C. Martinez / C. Martinez		10/1/15 0800			
Relinquished By/Removed From		Date/Time		Received By/Stored In		Date/Time			
T.R. [Signature]		10/1/15 0935		T.R. [Signature]		10-1-15 0935			
Relinquished By/Removed From		Date/Time		Received By/Stored In		Date/Time			
T.R. [Signature]		10-1-15 1800		Fed Ex		10-1-15			
Relinquished By/Removed From		Date/Time		Received By/Stored In		Date/Time			
[Signature]		10-2-15 9:35		[Signature]					
Relinquished By/Removed From		Date/Time		Received By/Stored In		Date/Time			
[Signature]									
Relinquished By/Removed From		Date/Time		Received By/Stored In		Date/Time			
[Signature]									
Relinquished By/Removed From		Date/Time		Received By/Stored In		Date/Time			
[Signature]									
FINAL SAMPLE DISPOSITION		Disposal Method		Disposed By		Date/Time			



280-74906 Chain of Custody



**JP1000**

**Appendix 5**  
**Data Validation Supporting Documentation**

## INORGANIC ANALYSIS DATA VALIDATION CHECKLIST

VALIDATION LEVEL:	A	B	<b>C</b>	D	E
PROJECT: 600-326			DATA PACKAGE: JP1000		
VALIDATOR: ELR		LAB: TAC		DATE: 10/15/15	
			SDG: JP1000		
ANALYSES PERFORMED					
<b>SW-846/ICP</b>	SW-846/GFAA	SW-846/Hg	SW-846 Cyanide		
SAMPLES/MATRIX					
J1V84C J1V847 J1V848 J1V849					
Soil					

## 1. DATA PACKAGE COMPLETENESS AND CASE NARRATIVE

Technical verification documentation present? ..... Yes **No** N/A

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

## 2. INSTRUMENT PERFORMANCE AND CALIBRATIONS (Levels D and E)

Initial calibrations performed on all instruments? ..... Yes No **N/A**

Initial calibrations acceptable? ..... Yes No **N/A**

ICP interference checks acceptable? ..... Yes No **N/A**

ICV and CCV checks performed on all instruments? ..... Yes No **N/A**

ICV and CCV checks acceptable? ..... Yes No **N/A**

Standards traceable? ..... Yes No **N/A**

Standards expired? ..... Yes No **N/A**

Calculation check acceptable? ..... Yes No **N/A**

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

## INORGANIC ANALYSIS DATA VALIDATION CHECKLIST

## 3. BLANKS (Levels B, C, D, and E)

ICB and CCB checks performed for all applicable analyses? (Levels D, E)..... Yes No N/A

ICB and CCB results acceptable? (Levels D, E) ..... Yes No N/A

Laboratory blanks analyzed? ..... Yes No N/A

Laboratory blank results acceptable?..... Yes No N/A

Field blanks analyzed? (Levels C, D, E) ..... Yes No N/A

Field blank results acceptable? (Levels C, D, E) ..... Yes No N/A

Transcription/calculation errors? (Levels D, E)..... Yes No N/A

Comments: 45. calcium, chromium, magnesium nickel - UJ

## 4. ACCURACY (Levels C, D, and E)

MS/MSD samples analyzed? ..... Yes No N/A

MS/MSD results acceptable? ..... Yes No N/A

MS/MSD standards NIST traceable? (Levels D, E) ..... Yes No N/A

MS/MSD standards expired? (Levels D, E) ..... Yes No N/A

LCS/BSS samples analyzed? ..... Yes No N/A

LCS/BSS results acceptable? ..... Yes No N/A

Standards traceable? (Levels D, E)..... Yes No N/A

Standards expired? (Levels D, E) ..... Yes No N/A

Transcription/calculation errors? (Levels D, E)..... Yes No N/A

Performance audit sample(s) analyzed? ..... Yes No N/A

Performance audit sample results acceptable?..... Yes No N/A

Comments: LCS - silicon (1990) - J

MS - antimony (5320) silicon (1690) - J all

NO PAH

**INORGANIC ANALYSIS DATA VALIDATION CHECKLIST****5. PRECISION (Levels C, D, and E)**

Duplicate RPD values acceptable? .....	<u>Yes</u>	No	N/A
Duplicate results acceptable? .....	<u>Yes</u>	No	N/A
MS/MSD standards NIST traceable? (Levels D, E) .....	Yes	No	<u>N/A</u>
MS/MSD standards expired? (Levels D, E) .....	Yes	No	<u>N/A</u>
Field duplicate RPD values acceptable? .....	<u>Yes</u>	No	N/A
Field split RPD values acceptable? .....	Yes	No	<u>N/A</u>
Transcription/calculation errors? (Levels D, E) .....	Yes	No	<u>N/A</u>

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**6. ICP QUALITY CONTROL (Levels D and E)**

ICP serial dilution samples analyzed? .....	Yes	No	N/A
ICP serial dilution %D values acceptable? .....	Yes	No	N/A
ICP post digestion spike required? .....	Yes	No	N/A
ICP post digestion spike values acceptable? .....	Yes	No	N/A
Standards traceable? .....	Yes	No	N/A
Standards expired? .....	Yes	No	N/A
Transcription/calculation errors? .....	Yes	No	N/A

Comments: \_\_\_\_\_

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**INORGANIC ANALYSIS DATA VALIDATION CHECKLIST****7. FURNACE AA QUALITY CONTROL (Levels D and E)**

Duplicate injections performed as required? .....	Yes	No	N/A
Duplicate injection %RSD values acceptable? .....	Yes	No	N/A
Analytical spikes performed as required? .....	Yes	No	N/A
Analytical spike recoveries acceptable? .....	Yes	No	N/A
Standards traceable? .....	Yes	No	N/A
Standards expired? .....	Yes	No	N/A
MSA performed as required? .....	Yes	No	N/A
MSA results acceptable? .....	Yes	No	N/A
Transcription/calculation errors? .....	Yes	No	N/A

Comments: \_\_\_\_\_

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**8. HOLDING TIMES (all levels)**

Samples properly preserved? .....	Yes	No	N/A
Sample holding times acceptable? .....	Yes	No	N/A

Comments: \_\_\_\_\_

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# INORGANIC ANALYSIS DATA VALIDATION CHECKLIST

## 9. RESULT QUANTITATION AND DETECTION LIMITS (all levels)

Results reported for all requested analyses? .....	<input checked="" type="radio"/> Yes	<input type="radio"/> No	<input type="radio"/> N/A
Results supported in the raw data? (Levels D, E) .....	<input type="radio"/> Yes	<input type="radio"/> No	<input checked="" type="radio"/> N/A
Samples properly prepared? (Levels D, E) .....	<input type="radio"/> Yes	<input type="radio"/> No	<input checked="" type="radio"/> N/A
Detection limits meet RDL? .....	<input checked="" type="radio"/> Yes	<input type="radio"/> No	<input type="radio"/> N/A
Transcription/calculation errors? (Levels D, E) .....	<input type="radio"/> Yes	<input type="radio"/> No	<input checked="" type="radio"/> N/A

Comments: \_\_\_\_\_

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**Appendix 6**  
**Additional Documentation Requested by Client**

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

### Method Blank - Batch: 280-297784

Method: 6010B  
Preparation: 3050B

Lab Sample ID: MB 280-297784/1-A  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/06/2015 1845  
Prep Date: 10/05/2015 1350  
Leach Date: N/A

Analysis Batch: 280-298187  
Prep Batch: 280-297784  
Leach Batch: N/A  
Units: mg/Kg

Instrument ID: MT\_025  
Lab File ID: 25B100615.asc  
Initial Weight/Volume: 1 g  
Final Weight/Volume: 100 mL

Analyte	Result	Qual	MDL	RL
Antimony	0.38	U	0.38	0.60
Arsenic	0.66	U	0.66	1.0
Barium	0.156	B	0.076	0.50
Beryllium	0.033	U	0.033	0.20
Boron	0.98	U	0.98	2.0
Cadmium	0.041	U	0.041	0.20
Calcium	20.98	B	14.1	50.0
Chromium	0.0960	B	0.058	0.20
Cobalt	0.10	U	0.10	1.0
Copper	0.22	U	0.22	1.0
Lead	0.27	U	0.27	0.50
Magnesium	6.33	B	3.7	20.0
Manganese	0.10	U	0.10	1.0
Molybdenum	0.26	U	0.26	2.0
Nickel	0.126	B	0.12	4.0
Potassium	41.0	U	41.0	300
Selenium	0.86	U	0.86	1.0
Silicon	5.7	U	5.7	10.0
Silver	0.16	U	0.16	0.20
Sodium	59.0	U	59.0	120
Vanadium	0.094	U	0.094	2.0

### Method Blank - Batch: 280-297784

Method: 6010B  
Preparation: 3050B

Lab Sample ID: MB 280-297784/1-A  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/07/2015 1557  
Prep Date: 10/05/2015 1350  
Leach Date: N/A

Analysis Batch: 280-298379  
Prep Batch: 280-297784  
Leach Batch: N/A  
Units: mg/Kg

Instrument ID: MT\_025  
Lab File ID: 25A100715.asc  
Initial Weight/Volume: 1 g  
Final Weight/Volume: 100 mL

Analyte	Result	Qual	MDL	RL
Aluminum	1.6	U	1.6	5.0
Iron	8.50		3.8	5.0
Zinc	0.797	B	0.40	1.0

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

### Lab Control Sample - Batch: 280-297784

Method: 6010B

Preparation: 3050B

Lab Sample ID: LCS 280-297784/2-A  
 Client Matrix: Solid  
 Dilution: 1.0  
 Analysis Date: 10/06/2015 1848  
 Prep Date: 10/05/2015 1350  
 Leach Date: N/A

Analysis Batch: 280-298187  
 Prep Batch: 280-297784  
 Leach Batch: N/A  
 Units: mg/Kg

Instrument ID: MT\_025  
 Lab File ID: 25B100615.asc  
 Initial Weight/Volume: 1 g  
 Final Weight/Volume: 100 mL

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Antimony	50.0	50.30	101	82 - 110	
Arsenic	100	100.0	100	85 - 110	
Barium	200	214.4	107	87 - 112	
Beryllium	5.00	5.05	101	84 - 114	
Boron	100	100.6	101	80 - 120	
Cadmium	10.0	10.55	105	87 - 110	
Calcium	5000	5205	104	82 - 114	
Chromium	20.0	20.68	103	84 - 114	
Cobalt	50.0	50.38	101	87 - 110	
Copper	25.0	26.70	107	88 - 110	
Lead	50.0	50.61	101	86 - 110	
Magnesium	5000	4993	100	90 - 110	
Manganese	50.0	50.20	100	88 - 110	
Molybdenum	100	105.6	106	86 - 110	
Nickel	50.0	51.72	103	87 - 110	
Potassium	5000	5282	106	89 - 110	
Selenium	200	195.1	98	83 - 110	
Silicon	1000	188.7	19	10 - 70	
Silver	5.00	5.23	105	87 - 114	
Sodium	5000	5507	110	90 - 112	
Vanadium	50.0	51.44	103	88 - 110	

### Lab Control Sample - Batch: 280-297784

Method: 6010B

Preparation: 3050B

Lab Sample ID: LCS 280-297784/2-A  
 Client Matrix: Solid  
 Dilution: 1.0  
 Analysis Date: 10/07/2015 1559  
 Prep Date: 10/05/2015 1350  
 Leach Date: N/A

Analysis Batch: 280-298379  
 Prep Batch: 280-297784  
 Leach Batch: N/A  
 Units: mg/Kg

Instrument ID: MT\_025  
 Lab File ID: 25A100715.asc  
 Initial Weight/Volume: 1 g  
 Final Weight/Volume: 100 mL

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Aluminum	200	198.2	99	82 - 116	
Iron	100	111.6	112	87 - 120	
Zinc	50.0	51.33	103	76 - 114	

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

### Matrix Spike - Batch: 280-297784

Method: 6010B

Preparation: 3050B

Lab Sample ID:	280-74906-1	Analysis Batch:	280-298187	Instrument ID:	MT_025
Client Matrix:	Solid	Prep Batch:	280-297784	Lab File ID:	25B100615.asc
Dilution:	1.0	Leach Batch:	N/A	Initial Weight/Volume:	1.193 g
Analysis Date:	10/06/2015 1858	Units:	mg/Kg	Final Weight/Volume:	100 mL
Prep Date:	10/05/2015 1350				
Leach Date:	N/A				

Analyte	Sample Result/Qual	Spike Amount	Result	% Rec.	Limit	Qual
Antimony	0.38 U	46.4	24.06	52	20 - 200	
Arsenic	3.5	92.8	83.40	86	76 - 111	
Barium	80.8	186	266.5	100	52 - 159	
Beryllium	0.28	4.64	4.36	88	72 - 105	
Boron	1.2 B	92.8	79.93	85	80 - 120	
Cadmium	0.14 B	9.28	8.62	91	40 - 130	
Calcium	3570	4640	8987	117	43 - 165	
Chromium	9.4	18.6	26.88	94	70 - 200	
Cobalt	8.1	46.4	47.39	85	72 - 106	
Copper	11.3	23.2	32.89	93	37 - 187	
Lead	6.0	46.4	45.98	86	70 - 200	
Magnesium	4260	4640	8688	95	64 - 145	
Manganese	307	46.4	373.5	144	40 - 200	4
Molybdenum	0.26 U	92.8	82.83	89	75 - 103	
Nickel	9.8	46.4	49.50	86	61 - 126	
Potassium	1700	4640	6398	101	56 - 172	
Selenium	0.85 U	186	156.2	84	76 - 104	
Silicon	325	928	473.3	16	20 - 200	N
Silver	0.16 U	4.64	4.25	92	75 - 141	
Sodium	192	4640	4836	100	78 - 111	
Vanadium	51.9	46.4	94.66	92	50 - 169	

### Matrix Spike - Batch: 280-297784

Method: 6010B

Preparation: 3050B

Lab Sample ID:	280-74906-1	Analysis Batch:	280-298379	Instrument ID:	MT_025
Client Matrix:	Solid	Prep Batch:	280-297784	Lab File ID:	25A100715.asc
Dilution:	1.0	Leach Batch:	N/A	Initial Weight/Volume:	1.193 g
Analysis Date:	10/07/2015 1609	Units:	mg/Kg	Final Weight/Volume:	100 mL
Prep Date:	10/05/2015 1350				
Leach Date:	N/A				

Analyte	Sample Result/Qual	Spike Amount	Result	% Rec.	Limit	Qual
Aluminum	7590	186	10160	1382	50 - 200	4
Iron	20700	92.8	21020	296	70 - 200	4
Zinc	41.8	46.4	83.16	89	70 - 200	

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1  
Sdg Number: JP1000

**Duplicate - Batch: 280-297784**

**Method: 6010B**  
**Preparation: 3050B**

Lab Sample ID: 280-74906-1  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/06/2015 1855  
Prep Date: 10/05/2015 1350  
Leach Date: N/A

Analysis Batch: 280-298187  
Prep Batch: 280-297784  
Leach Batch: N/A  
Units: mg/Kg

Instrument ID: MT\_025  
Lab File ID: 25B100615.asc  
Initial Weight/Volume: 1.129 g  
Final Weight/Volume: 100 mL

Analyte	Sample Result/Qual	Result	RPD	Limit	Qual
Antimony	0.38 U	0.37	NC	40	U
Arsenic	3.5	3.86	9	30	
Barium	80.8	97.17	18	30	
Beryllium	0.28	0.279	1	30	
Boron	1.2 B	1.20	0.3	30	B
Cadmium	0.14 B	0.121	12	30	B
Calcium	3570	3552	0.5	30	
Chromium	9.4	10.06	6	40	
Cobalt	8.1	7.82	3	30	
Copper	11.3	11.12	1	30	
Lead	6.0	6.32	6	40	
Magnesium	4260	4246	0.3	30	
Manganese	307	362.5	17	40	
Molybdenum	0.26 U	0.25	NC	30	U
Nickel	9.8	10.28	4	30	
Potassium	1700	1751	3	40	
Selenium	0.85 U	0.84	NC	30	U
Silicon	325	330.0	1	40	
Silver	0.16 U	0.16	NC	30	U
Sodium	192	194.7	1	30	
Vanadium	51.9	51.33	1	30	

**Duplicate - Batch: 280-297784**

**Method: 6010B**  
**Preparation: 3050B**

Lab Sample ID: 280-74906-1  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/07/2015 1607  
Prep Date: 10/05/2015 1350  
Leach Date: N/A

Analysis Batch: 280-298379  
Prep Batch: 280-297784  
Leach Batch: N/A  
Units: mg/Kg

Instrument ID: MT\_025  
Lab File ID: 25A100715.asc  
Initial Weight/Volume: 1.129 g  
Final Weight/Volume: 100 mL

Analyte	Sample Result/Qual	Result	RPD	Limit	Qual
Aluminum	7590	7843	3	40	
Iron	20700	20470	1	40	
Zinc	41.8	42.70	2	40	

Date: 19 August 2015  
To: Washington Closure Hanford Inc. (technical representative)  
From: ELR Consulting  
Project: 100-IU-2 & 100-IU-6 Remaining Waste Sites – Soil Full Protocol - Waste Site 600-326  
Subject: Pesticide - Data Package No. JP1000-TAL

## **INTRODUCTION**

This memo presents the results of data validation on Data Package No. JP1000 prepared by TestAmerica Laboratories (TAL). A list of samples validated along with the analyses reported and the method of analysis is provided in the following table.

Sample ID	Sample Date	Media	Validation	Analyte
J1V846	10/1/15	Soil	C	See note 1
J1V847	10/1/15	Soil	C	See note 1
J1V848	10/1/15	Soil	C	See note 1

1 – Pesticides by 8081B.

Data validation was conducted in accordance with the Washington Closure Hanford (WCH) validation statement of work and the 100 Area Remedial Action Sampling and Analysis Plan (DOE/RL-96-22, September 2009). Appendices 1 through 6 provide the following information as indicated below:

- Appendix 1. Glossary of Data Reporting Qualifiers
- Appendix 2. Summary of Data Qualification
- Appendix 3. Annotated Laboratory Reports
- Appendix 4. Laboratory Narrative and Chain-of-Custody Documentation
- Appendix 5. Data Validation Supporting Documentation
- Appendix 6. Additional Documentation Requested by Client

## **DATA QUALITY OBJECTIVES**

### **Holding Times**

Analytical holding times were assessed to ascertain whether the holding time requirements were met by the laboratory. The holding time requirements are as follows: Samples must be extracted within 14 days of the date of sample collection and analyzed within 40 days from the date of extraction.

If holding times are exceeded, but not by greater than two times the limit, all associated sample results are qualified as estimates and flagged "J" for detects and "UJ" for non-detects. If holding times are exceeded by greater than two times the limit, all

associated detectable sample results are qualified as estimates and flagged "J" and all non-detects are rejected and flagged "UR".

All holding times were acceptable.

## **Method Blank**

Method blank analyses are performed to determine the extent of laboratory contamination introduced through sampling, sample preparation or analysis. At least one method blank analysis must be conducted for every 20 samples. Method blanks should not contain target compounds at a concentration greater than required quantitation limit (RQL). If target compounds are present, sample results less than five times the blank concentration are qualified as undetected and flagged "U". If the sample result is less than five times the blank concentration and less than RQL, the result is qualified as undetected and elevated to the RQL.

All method blank results were acceptable.

## Field Blanks

No field blanks were submitted for analysis.

## **Accuracy**

### Matrix Spike & Laboratory Control Sample

Matrix spike (MS) and laboratory control sample (LCS) analyses are used to assess the analytical accuracy of the reported data. The matrix spike is used to assess the effect of the matrix on the ability to accurately quantify sample concentrations. Recoveries must fall within the range of 50% to 150%. If spike recoveries are outside control limits, detected sample results less than five times the spike concentration are qualified as estimates and flagged "J". Non-detected sample results with spike recoveries outside control limits are qualified as estimates and flagged "UJ". Sample results greater than five times the spike concentration require no qualification.

Due to the lack of a matrix spike, matrix spike duplicate or LCS analysis, all toxaphene results were qualified as estimates and flagged "J".

Due to a matrix spike recovery outside QC limits (49%), all endrin aldehyde results were qualified as estimates and flagged "J".

All other accuracy results were acceptable.

## Surrogate Recovery

The analysis of surrogate compounds provides a measure of performance for individual samples. Matrix-specific surrogate compound recovery control windows have been established by the laboratory. When a surrogate compound recovery is outside the control window, all positively identified target compounds associated with the unacceptable surrogate recoveries are qualified as estimates and flagged "J". Non-detected compounds with surrogate recoveries less than the lower control limit are qualified as having an estimated detection limit and flagged "UJ". Non-detected compounds with surrogate recoveries above the upper control limit require no qualification.

All surrogate results were acceptable.

## **Precision**

### Matrix Spike/Matrix Spike Duplicate Samples

Matrix spike/matrix spike duplicate results provide matrix-specific information on the precision of the method for specific target compound classes. Precision is expressed as the relative percent difference (RPD) between the recoveries of duplicate matrix spike analyses performed on a sample. For soil samples, results must be within RPD limits of plus/minus 30%. If RPD values are out of specification and the sample concentration is less than five times the spike concentration, all associated detected sample results are qualified as estimates and flagged "J". If RPD values are out of specification and the sample concentration is greater than five times the spike concentration, no qualification is required.

Due to the lack of a matrix spike and matrix spike duplicate analysis, all toxaphene results were qualified as estimates and flagged "J".

All other precision results were acceptable.

### Field Duplicate Samples

One set of field duplicates (J1V846/J1V848) were submitted for analysis. Field duplicates are compared using the same criteria as for laboratory duplicates. All field duplicate results were acceptable.

## **Analytical Detection Levels**

Reported analytical detection levels are compared against the 100 Area RQLs to ensure that laboratory detection levels meet the required criteria. All results met the RQL.



## **Completeness**

Data Package No. JP1000 was submitted for validation and verified for completeness. Completeness is based on the percentage of data determined to be valid (i.e., not rejected). The completion percentage was 100%.

## **MAJOR DEFICIENCIES**

None found.

## **MINOR DEFICIENCIES**

The following minor deficiencies were noted:

- Due to the lack of a matrix spike, matrix spike duplicate or LCS analysis, all toxaphene results were qualified as estimates and flagged "J".
- Due to a matrix spike recovery outside QC limits (49%), all endrin aldehyde results were qualified as estimates and flagged "J".

Data flagged "J" indicates that the associated concentration is an estimate, but under the WCH statement of work, the data may be usable for decision-making purposes. All other validated results are considered accurate within the standard error associated with the methods.

## **REFERENCES**

Washington Closure Hanford Contract #S00W307A00 (March 2008), *Data Validation Services*, March 2008.

DOE/RL-96-22, Rev. 5, *100 Area Remedial Action Sampling and Analysis Plan*, U.S. Department of Energy, September 2009.

**Appendix 1**  
**Glossary of Data Reporting Qualifiers**

Qualifiers which may be applied by data validators in compliance with the procedures herein are as follows:

- U - Indicates the compound or analyte was analyzed for and not detected in the sample. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory.
- UJ - Indicates the compound or analyte was analyzed for and not detected in the sample. Due to a minor QC deficiency identified during the data validation, the associated quantitation limit is an estimate.
- J - Indicates the compound or analyte was analyzed for and detected. Due to a minor QC deficiency identified during the data validation, the associated quantitation limit is an estimate.
- R - Indicates the compound or analyte was analyzed for, detected, and due to an identified major QC deficiency, the data are unusable.
- UR - Indicates the compound or analyte was analyzed for and not detected in the sample. Additionally, the data is unusable due to an identified major QC deficiency.
- NJ - Indicates presumptive evidence of a compound at an estimated value. The data may not be valid for some specific applications (i.e., usable for decision-making purposes).
- N - Indicates presumptive evidence of a compound. The data may not be valid for some specific applications (i.e., usable for decision-making purposes).

**Appendix 2**  
**Summary of Data Qualification**

# PESTICIDE DATA QUALIFICATION SUMMARY\*

<b>SDG: JP1000</b>	<b>REVIEWER: ELR</b>	<b>Project: 600-326</b>	<b>PAGE <u>1</u> OF <u>1</u></b>
<b>COMPOUND</b>	<b>QUALIFIER</b>	<b>SAMPLES AFFECTED</b>	<b>REASON</b>
Toxaphene	J	All	No MS/MSD/LCS analysis
Endrin aldehyde	J	All	MS recovery

\* - The Qualified Data Summary Table includes laboratory applied "U" qualifiers not specifically identified here. The laboratory applied "U" qualifiers are included to minimize misinterpretation of results contained in the table.

**Appendix 3**  
**Annotated Laboratory Reports**

# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1  
Sdg Number: JP1000

Client Sample ID: J1V846

Lab Sample ID: 280-74906-1  
Client Matrix: Solid

% Moisture: 9.7

Date Sampled: 10/01/2015 0755  
Date Received: 10/02/2015 0935

## 8081A Organochlorine Pesticides (GC)

Analysis Method: 8081A  
Prep Method: 3550C  
Dilution: 1.0  
Analysis Date: 10/07/2015 1854  
Prep Date: 10/04/2015 0934

Analysis Batch: 280-298206  
Prep Batch: 280-297790

Instrument ID: SGC\_C  
Initial Weight/Volume: 31.6 g  
Final Weight/Volume: 10 mL  
Injection Volume: 1. uL  
Result Type: PRIMARY

Analyte	DryWt Corrected: Y	Result (ug/Kg)	Qualifier	MDL	RL
4,4'-DDD		0.57	UN	0.57	1.8
4,4'-DDE		0.25	UN	0.25	1.8
4,4'-DDT		0.62	UN	0.62	1.8
Aldrin		0.26	U	0.26	1.7
alpha-BHC		0.22	U	0.22	1.7
beta-BHC		0.70	UN	0.70	1.7
delta-BHC		0.42	UN	0.42	1.7
gamma-BHC (Lindane)		0.49	U	0.49	1.7
Heptachlor		0.22	U	0.22	1.7
Heptachlor epoxide		0.45	U	0.45	1.7
Endosulfan I		0.18	UN	0.18	1.7
Endosulfan II		0.30	UN	0.30	1.8
Endosulfan sulfate		0.29	UN	0.29	1.8
Endrin		0.32	U	0.32	1.8
Endrin aldehyde		0.18	U J	0.18	1.8
Endrin ketone		0.51	UN	0.51	1.8
gamma-Chlordane		0.28	UN	0.28	1.8
Methoxychlor		0.47	UN	0.47	3.5
alpha-Chlordane		0.34	UN	0.34	1.8
Dieldrin		0.22	UN	0.22	1.8
Toxaphene		17	U J	17	170

Surrogate	%Rec	Qualifier	Acceptance Limits
Tetrachloro-m-xylene	75		59 - 115
Decachlorobiphenyl	72		63 - 124

10/12/15

# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1  
Sdg Number: JP1000

Client Sample ID: J1V847

Lab Sample ID: 280-74906-2  
Client Matrix: Solid

% Moisture: 11.5

Date Sampled: 10/01/2015 0720  
Date Received: 10/02/2015 0935

## 8081A Organochlorine Pesticides (GC)

Analysis Method: 8081A  
Prep Method: 3550C  
Dilution: 1.0  
Analysis Date: 10/07/2015 1946  
Prep Date: 10/04/2015 0934

Analysis Batch: 280-298206  
Prep Batch: 280-297790

Instrument ID: SGC\_C  
Initial Weight/Volume: 31.6 g  
Final Weight/Volume: 10 mL  
Injection Volume: 1 uL  
Result Type: PRIMARY

Analyte	DryWt Corrected: Y	Result (ug/Kg)	Qualifier	MDL	RL
4,4'-DDD		0.59	U	0.59	1.8
4,4'-DDE		0.59	JY	0.26	1.8
4,4'-DDT		0.63	U	0.63	1.8
Aldrin		0.27	U	0.27	1.8
alpha-BHC		0.23	U	0.23	1.8
beta-BHC		0.71	U	0.71	1.8
delta-BHC		0.43	U	0.43	1.8
gamma-BHC (Lindane)		0.50	U	0.50	1.8
Heptachlor		0.23	U	0.23	1.8
Heptachlor epoxide		0.46	U	0.46	1.8
Endosulfan I		0.19	U	0.19	1.8
Endosulfan II		0.31	U	0.31	1.8
Endosulfan sulfate		0.30	U	0.30	1.8
Endrin		0.33	U	0.33	1.8
Endrin aldehyde		0.18	U J	0.18	1.8
Endrin ketone		0.52	U	0.52	1.8
gamma-Chlordane		0.29	U	0.29	1.8
Methoxychlor		0.48	U	0.48	3.5
alpha-Chlordane		0.35	U	0.35	1.8
Dieldrin		0.23	U	0.23	1.8
Toxaphene		17	U J	17	180

Surrogate	%Rec	Qualifier	Acceptance Limits
Tetrachloro-m-xylene	67		59 - 115
Decachlorobiphenyl	71		63 - 124

✓ 10/17/11



# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1  
Sdg Number: JP1000

Client Sample ID: J1V848

Lab Sample ID: 280-74906-3

Client Matrix: Solid

% Moisture: 8.5

Date Sampled: 10/01/2015 0755

Date Received: 10/02/2015 0935

## 8081A Organochlorine Pesticides (GC)

Analysis Method: 8081A  
Prep Method: 3550C  
Dilution: 1.0  
Analysis Date: 10/07/2015 2004  
Prep Date: 10/04/2015 0934

Analysis Batch: 280-298206  
Prep Batch: 280-297790

Instrument ID: SGC\_C  
Initial Weight/Volume: 30.0 g  
Final Weight/Volume: 10 mL  
Injection Volume: 1 uL  
Result Type: PRIMARY

Analyte	DryWt Corrected: Y	Result (ug/Kg)	Qualifier	MDL	RL
4,4'-DDD		0.60	U	0.60	1.9
4,4'-DDE		0.26	U	0.26	1.9
4,4'-DDT		0.64	U	0.64	1.9
Aldrin		0.27	U	0.27	1.8
alpha-BHC		0.23	U	0.23	1.8
beta-BHC		0.73	U	0.73	1.8
delta-BHC		0.44	U	0.44	1.8
gamma-BHC (Lindane)		0.51	U	0.51	1.8
Heptachlor		0.23	U	0.23	1.8
Heptachlor epoxide		0.47	U	0.47	1.8
Endosulfan I		0.19	U	0.19	1.8
Endosulfan II		0.31	U	0.31	1.9
Endosulfan sulfate		0.30	U	0.30	1.9
Endrin		0.33	U	0.33	1.9
Endrin aldehyde		0.19	U J	0.19	1.9
Endrin ketone		0.53	U	0.53	1.9
gamma-Chlordane		0.29	U	0.29	1.9
Methoxychlor		0.49	U	0.49	3.6
alpha-Chlordane		0.35	U	0.35	1.9
Dieldrin		0.23	U	0.23	1.9
Toxaphene		17	U J	17	180

Surrogate	%Rec	Qualifier	Acceptance Limits
Tetrachloro-m-xylene	77		59 - 115
Decachlorobiphenyl	68		63 - 124

16/17/15

## **Appendix 4**

### **Laboratory Narrative and Chain-of-Custody Documentation**

## CASE NARRATIVE

Client: Washington Closure Hanford

Project: WASHINGTON CLOSURE HANFORD

Job Number: 280-74906-1

SDG #: JP1000

SAF#: RC-232

Date SDG Closed: October 2, 2015

Data Deliverable: 7 Day / Summary

<u>CLIENT ID</u>	<u>LAB ID</u>	<u>ANALYSES REQUESTED</u>	<u>ANALYSES PERFORMED</u>
J1V846	280-74906-1	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V847	280-74906-2	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V848	280-74906-3	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V849	280-74906-4	6010	6010B

I certify that this data package is in compliance with the SOW, both technically and for completeness, for other than the conditions detailed in this Case Narrative. Release of the data contained in this hard copy data package has been authorized by the Laboratory Manager or a designee, as verified by the signature on the Report Cover.

With exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. All laboratory quality control samples analyzed in conjunction with the samples in this project were within established control limits, with any exceptions noted. Calculations are performed before rounding to avoid round-off errors in calculated results.

This report includes reporting limits (RLs) less than TestAmerica Denver's practical quantitation limits. These reporting limits are being used specifically at the client's request to meet the needs of this project. Please note that data are not normally reported to these levels without qualification, since they are inherently less reliable and potentially less defensible than required by the current NELAC standards.

The results, RLs and MDLs included in this report have been adjusted for dry weight, as appropriate.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

### RECEIPT

The samples were received on 10/2/2015 9:35 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperature of the cooler at receipt was 3.3° C.

### GC SEMIVOLATILES - SW846 8081A - Pesticides

The organic prep laboratory noted that the samples presented in this report required a Florisil clean-up to reduce matrix interferences.

The laboratory noted that the samples presented in this report required a mercury clean-up to reduce matrix interferences caused by sulfur.

The RPD between the primary and confirmation columns exceeded 40% for 4,4'-DDE in sample J1V847. The higher of the two values has been reported. The result has been flagged with a "Y".

The MS/MSD performed on sample J1V846 exhibited spike compound recoveries outside the control limits, and the associated sample results have been flagged "N". In addition, the RPD limit was exceeded for 4,4'-DDD and 4,4'-DDE. The acceptable LCS analysis data indicated that the analytical system was operating within control; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

### HPLC - SW846 8310 - PAHs

The RPD between the primary and confirmation columns exceeded 40% for Benzo[a]anthracene and Phenanthrene in sample J1V847. The lower of the two values has been reported, as matrix interference is evident on both columns. The results have been flagged with an "X".

The MS/MSD performed on sample J1V847 exhibited percent recoveries outside the control limits for Chrysene, and the associated sample result has been flagged "N". The acceptable LCS analysis data indicated that the analytical system was operating within control; therefore, corrective action is deemed unnecessary.

Continuing Calibration Verification (CCV) standards associated with samples in analysis batch 280-298048 exhibited %Difference (%D) values >15%, biased high, for Benzo[g,h,i]perylene, Dibenzo[a,h]anthracene and Fluoranthene. The samples associated with these CCVs are either non-detect or less than the reporting limit; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

#### **TOTAL METALS - SW846 6010B**

Serial dilution of a digestate in batch 280-297784 indicates that physical and chemical interferences are present for Iron and Zinc. Results have been flagged with an "X".

Low levels of Barium, Calcium, Chromium, Magnesium and Nickel are present in the method blank associated with batch 280-297784. Because the concentrations in the method blank are not present at levels greater than half the reporting limit, corrective action is deemed unnecessary.

Zinc is present in the method blank associated with batch 280-297784 at 0.797 mg/kg, which is greater than half the project specific reporting limit (PSRL) of 1 mg/kg. TestAmerica's practical quantitation limit (PQL) for Zinc is 3 mg/kg. The laboratory cannot maintain system cleanliness at this low level; therefore, corrective action is not initiated. It can be noted that the concentration found in the method blank is less than half of the laboratory standard PQL, and with the exception of 'blank' sample J1V849, the associated sample amounts are greater than twenty times the method blank concentration.

Iron, a common laboratory contaminant, is present at a level greater than the reporting limit in the method blank associated with batch 280-297784. As the associated sample amounts are greater than twenty times the method blank concentration, corrective action is deemed unnecessary.

It can be noted that the sample amount was greater than four times the spike amount for Aluminum, Iron and Manganese in the Matrix Spike performed on sample J1V846; therefore, control limits are not applicable.

Silicon was recovered outside the control limits in the Matrix Spike performed on sample J1V846, and the associated sample result has been flagged "N". There is no indication that the analytical system was operating out of control, and method accuracy has been verified by the acceptable LCS analysis data; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

#### **GENERAL CHEMISTRY - SW846 9056M - ANIONS**

Each sample is analyzed to achieve the lowest possible reporting limits within the constraints of the method. Due to high constituent concentration, the Sulfate analysis of sample J1V847 had to be performed at a dilution, and the associated result has been flagged with a "D". The reporting limit has been adjusted relative to the dilution required.

Sulfate is present in the method blank associated with batch 280-297863 at 7.21 mg/kg, which is greater than the project specific reporting limit (PSRL) of 5 mg/kg. TestAmerica's practical quantitation limit (PQL) for Sulfate is 50 mg/kg. The laboratory cannot maintain system cleanliness at this low level; therefore, corrective action is not initiated. It can be noted that the concentration found in the method blank is less than half of the laboratory standard PQL.

The Matrix Spike performed on sample J1V846 exhibited the percent recovery outside the control limits for Fluoride, and the associated sample result has been flagged "N". There is no indication that the analytical system was operating out of control, and method accuracy has been verified by the acceptable LCS analysis data; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.



**Appendix 5**  
**Data Validation Supporting Documentation**

## PCB DATA VALIDATION CHECKLIST

VALIDATION LEVEL:	A	B	<b>C</b>	D	E
PROJECT: 600-326			DATA PACKAGE: JP1000		
VALIDATOR: ELR		LAB: TAL		DATE: 10/15/15	
			SDG: JP1000		
ANALYSES PERFORMED					
<b>SW-846 8081</b>	SW-846 8081 (TCLP)	SW-846 8082	SW-846 8081 (TCLP)		
SAMPLES/MATRIX					
J1V846 J1V847 J1V848					
Soil					

## 1. DATA PACKAGE COMPLETENESS AND CASE NARRATIVE

Technical verification documentation present? ..... Yes **No** N/A

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

## 2. INSTRUMENT PERFORMANCE AND CALIBRATIONS (Levels D and E)

Initial calibrations acceptable? ..... Yes No **N/A**

Continuing calibrations acceptable? ..... Yes No **N/A**

Standards traceable? ..... Yes No **N/A**

Standards expired? ..... Yes No **N/A**

Calculation check acceptable? ..... Yes No **N/A**

DDT and endrin breakdowns acceptable? ..... Yes No **N/A**

Comments: \_\_\_\_\_

\_\_\_\_\_

## PCB DATA VALIDATION CHECKLIST

## 3. BLANKS (Levels B, C, D, and E)

Calibration blanks analyzed? (Levels D, E) ..... Yes No N/A  
 Calibration blank results acceptable? (Levels D, E) ..... Yes No N/A  
 Laboratory blanks analyzed? ..... Yes No N/A  
 Laboratory blank results acceptable? ..... Yes No N/A  
 Field/trip blanks analyzed? (Levels C, D, E) ..... Yes No N/A  
 Field/trip blank results acceptable? (Levels C, D, E) ..... Yes No N/A  
 Transcription/calculation errors? (Levels D, E) ..... Yes No N/A

Comments: no FB

## 4. ACCURACY (Levels C, D, and E)

Surrogates analyzed? ..... Yes No N/A  
 Surrogate recoveries acceptable? ..... Yes No N/A  
 Surrogates traceable? (Levels D, E) ..... Yes No N/A  
 Surrogates expired? (Levels D, E) ..... Yes No N/A  
 MS/MSD samples analyzed? ..... Yes No N/A  
 MS/MSD results acceptable? ..... Yes No N/A  
 MS/MSD standards NIST traceable? (Levels D, E) ..... Yes No N/A  
 MS/MSD standards expired? (Levels D, E) ..... Yes No N/A  
 LCS/BSS samples analyzed? ..... Yes No N/A  
 LCS/BSS results acceptable? ..... Yes No N/A  
 Standards traceable? (Levels D, E) ..... Yes No N/A  
 Standards expired? (Levels D, E) ..... Yes No N/A  
 Transcription/calculation errors? (Levels D, E) ..... Yes No N/A  
 Performance audit sample(s) analyzed? ..... Yes No N/A  
 Performance audit sample results acceptable? ..... Yes No N/A

Comments: MS-endrin aldehyde (49%) - Tallno tox MS/MSD/LCSno PAS



## PCB DATA VALIDATION CHECKLIST

## 5. PRECISION (Levels C, D, and E)

Duplicate RPD values acceptable? ..... ☒ Yes No N/A  
 Duplicate results acceptable? ..... ☒ Yes No N/A  
 MS/MSD standards NIST traceable? (Levels D, E) ..... ☒ Yes No N/A  
 MS/MSD standards expired? (Levels D, E) ..... ☒ Yes No N/A  
 Field duplicate RPD values acceptable? ..... ☒ Yes No N/A  
 Field split RPD values acceptable? ..... ☒ Yes No N/A  
 Transcription/calculation errors? (Levels D, E) ..... ☒ Yes No N/A

Comments: \_\_\_\_\_

no for ms/msd - Jall

## 6. SYSTEM PERFORMANCE (Levels D and E)

Chromatographic performance acceptable? ..... ☒ Yes No N/A  
 Positive results resolved acceptably? ..... ☒ Yes No N/A

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

## 7. HOLDING TIMES (all levels)

Samples properly preserved? ..... ☒ Yes No N/A  
 Sample holding times acceptable? ..... ☒ Yes No N/A

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**PCB DATA VALIDATION CHECKLIST****8. COMPOUND IDENTIFICATION, QUANTITATION, AND DETECTION LIMITS (all levels)**

Compound identification acceptable? (Levels D, E) .....	Yes	No	N/A
Compound quantitation acceptable? (Levels D, E) .....	Yes	No	N/A
Results reported for all requested analyses? .....	Yes	No	N/A
Results supported in the raw data? (Levels D, E) .....	Yes	No	N/A
Samples properly prepared? (Levels D, E) .....	Yes	No	N/A
Detection limits meet RDL? .....	Yes	No	N/A
Transcription/calculation errors? (Levels D, E) .....	Yes	No	N/A

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**9. SAMPLE CLEANUP (Levels D and E)**

Fluorilcil ® (or other absorbent) cleanup performed? .....	Yes	No	N/A
Lot check performed? .....	Yes	No	N/A
Check recoveries acceptable? .....	Yes	No	N/A
GPC cleanup performed? .....	Yes	No	N/A
GPC check performed? .....	Yes	No	N/A
GPC check recoveries acceptable? .....	Yes	No	N/A
GPC calibration performed? .....	Yes	No	N/A
GPC calibration check performed? .....	Yes	No	N/A
GPC calibration check retention times acceptable? .....	Yes	No	N/A
Check/calibration materials traceable? .....	Yes	No	N/A
Check/calibration materials Expired? .....	Yes	No	N/A
Analytical batch QC given similar cleanup? .....	Yes	No	N/A
Transcription/Calculation Errors? .....	Yes	No	N/A

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**Appendix 6**  
**Additional Documentation Requested by Client**

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Method Blank - Batch: 280-297790

Method: 8081A  
Preparation: 3550C

Lab Sample ID: MB-280-297790/1-A  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/07/2015 2021  
Prep Date: 10/04/2015 0934  
Leach Date: N/A

Analysis Batch: 280-298206  
Prep Batch: 280-297790  
Leach Batch: N/A  
Units: ug/Kg

Instrument ID: SGC\_C  
Lab File ID: 10070036.D  
Initial Weight/Volume: 30.4 g  
Final Weight/Volume: 10 mL  
Injection Volume: 1 uL  
Column ID: PRIMARY

Analyte	Result	Qual	MDL	RL
4,4'-DDD	0.54	U	0.54	1.7
4,4'-DDE	0.23	U	0.23	1.7
4,4'-DDT	0.58	U	0.58	1.7
Aldrin	0.25	U	0.25	1.6
alpha-BHC	0.21	U	0.21	1.6
beta-BHC	0.66	U	0.66	1.6
delta-BHC	0.40	U	0.40	1.6
gamma-BHC (Lindane)	0.46	U	0.46	1.6
Heptachlor	0.21	U	0.21	1.6
Heptachlor epoxide	0.42	U	0.42	1.6
Endosulfan I	0.17	U	0.17	1.6
Endosulfan II	0.28	U	0.28	1.7
Endosulfan sulfate	0.27	U	0.27	1.7
Endrin	0.30	U	0.30	1.7
Endrin aldehyde	0.17	U	0.17	1.7
Endrin ketone	0.48	U	0.48	1.7
gamma-Chlordane	0.26	U	0.26	1.7
Methoxychlor	0.44	U	0.44	3.3
alpha-Chlordane	0.32	U	0.32	1.7
Dieldrin	0.21	U	0.21	1.7
Toxaphene	16	U	16	160

Surrogate	% Rec	Acceptance Limits
Tetrachloro-m-xylene	77	59 - 115
Decachlorobiphenyl	80	63 - 124

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1  
Sdg Number: JP1000

Lab Control Sample - Batch: 280-297790

Method: 8081A  
Preparation: 3550C

Lab Sample ID:	LCS 280-297790/2-A	Analysis Batch:	280-298206	Instrument ID:	SGC_C
Client Matrix:	Solid	Prep Batch:	280-297790	Lab File ID:	10070030.D
Dilution:	1.0	Leach Batch:	N/A	Initial Weight/Volume:	32.5 g
Analysis Date:	10/07/2015 1836	Units:	ug/Kg	Final Weight/Volume:	10 mL
Prep Date:	10/04/2015 0934			Injection Volume:	1 uL
Leach Date:	N/A			Column ID:	PRIMARY

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
4,4'-DDD	15.4	13.5	88	69 - 126	
4,4'-DDE	15.4	13.3	86	71 - 116	
4,4'-DDT	15.4	14.2	92	67 - 132	
Aldrin	15.4	12.7	82	69 - 116	
alpha-BHC	15.4	12.8	83	65 - 122	
beta-BHC	15.4	10.2	66	62 - 121	
delta-BHC	15.4	12.6	82	67 - 122	
gamma-BHC (Lindane)	15.4	12.6	82	66 - 120	
Heptachlor	15.4	13.0	85	61 - 126	
Heptachlor epoxide	15.4	13.3	87	71 - 119	
Endosulfan I	15.4	12.0	78	67 - 115	
Endosulfan II	15.4	13.0	84	69 - 120	
Endosulfan sulfate	15.4	13.7	89	69 - 126	
Endrin	15.4	14.0	91	69 - 129	
Endrin aldehyde	15.4	11.8	77	41 - 128	
Endrin ketone	15.4	13.2	86	70 - 125	
gamma-Chlordane	15.4	13.1	85	69 - 122	
Methoxychlor	15.4	14.5	94	65 - 139	
alpha-Chlordane	15.4	13.0	84	71 - 118	
Dieldrin	15.4	13.4	87	71 - 120	
Surrogate		% Rec		Acceptance Limits	
Tetrachloro-m-xylene		76		59 - 115	
Decachlorobiphenyl		85		63 - 124	

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

### Matrix Spike/

Matrix Spike Duplicate Recovery Report - Batch: 280-297790

Method: 8081A

Preparation: 3550C

MS Lab Sample ID: 280-74906-1  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/07/2015 1911  
Prep Date: 10/04/2015 0934  
Leach Date: N/A

Analysis Batch: 280-298206  
Prep Batch: 280-297790  
Leach Batch: N/A

Instrument ID: SGC\_C  
Lab File ID: 10070032.D  
Initial Weight/Volume: 31.4 g  
Final Weight/Volume: 10 mL  
Injection Volume: 1 uL  
Column ID: PRIMARY

MSD Lab Sample ID: 280-74906-1  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/07/2015 1929  
Prep Date: 10/04/2015 0934  
Leach Date: N/A

Analysis Batch: 280-298206  
Prep Batch: 280-297790  
Leach Batch: N/A

Instrument ID: SGC\_C  
Lab File ID: 10070033.D  
Initial Weight/Volume: 30.4 g  
Final Weight/Volume: 10 mL  
Injection Volume: 1 uL  
Column ID: PRIMARY

Analyte	% Rec.		Limit	RPD	RPD Limit	MS Qual	MSD Qual
	MS	MSD					
4,4'-DDD	55	67	69 - 126	22	20	N	N *
4,4'-DDE	65	76	71 - 116	19	15	N	*
4,4'-DDT	66	77	67 - 132	19	29	N	
Aldrin	72	79	69 - 116	13	50		
alpha-BHC	67	75	65 - 122	16	17		
beta-BHC	56	60	62 - 121	11	17	N	N
delta-BHC	65	74	67 - 122	16	19	N	
gamma-BHC (Lindane)	66	75	66 - 120	15	24		
Heptachlor	71	81	61 - 126	17	18		
Heptachlor epoxide	72	78	71 - 119	12	18		
Endosulfan I	59	66	67 - 115	14	26	N	N
Endosulfan II	65	74	69 - 120	17	20	N	
Endosulfan sulfate	63	72	69 - 126	16	22	N	
Endrin	70	81	69 - 129	18	30		
Endrin aldehyde	49	55	41 - 128	14	29		
Endrin ketone	63	71	70 - 125	15	20	N	
gamma-Chlordane	65	74	69 - 122	16	21	N	
Methoxychlor	62	70	65 - 139	16	23	N	
alpha-Chlordane	62	71	71 - 118	16	18	N	
Dieldrin	65	76	71 - 120	19	25	N	
Surrogate	MS % Rec		MSD % Rec	Acceptance Limits			
Tetrachloro-m-xylene	76		74	59 - 115			
Decachlorobiphenyl	68		64	63 - 124			

Date: 19 October 2015  
To: Washington Closure Hanford Inc. (technical representative)  
From: ELR Consulting  
Project: 100-IU-2 & 100-IU-6 Remaining Waste Sites – Soil Full Protocol - Waste Site 600-326  
Subject: Polyaromatic Hydrocarbon - Data Package No. JP1000-TAL

## **INTRODUCTION**

This memo presents the results of data validation on Data Package No. JP1000 prepared by TestAmerica Laboratories (TAL). A list of samples validated along with the analyses reported and the method of analysis is provided in the following table.

<b>Sample ID</b>	<b>Sample Date</b>	<b>Media</b>	<b>Validation</b>	<b>Analyte</b>
J1V846	10/1/15	Soil	C	See note 1
J1V847	10/1/15	Soil	C	See note 1
J1V848	10/1/15	Soil	C	See note 1

1 – Polyaromatic hydrocarbons by 8310.

Data validation was conducted in accordance with the Washington Closure Hanford (WCH) validation statement of work and the 100 Area Remedial Action Sampling and Analysis Plan (DOE/RL-96-22, September 2009). Appendices 1 through 6 provide the following information as indicated below:

- Appendix 1. Glossary of Data Reporting Qualifiers
- Appendix 2. Summary of Data Qualification
- Appendix 3. Annotated Laboratory Reports
- Appendix 4. Laboratory Narrative and Chain-of-Custody Documentation
- Appendix 5. Data Validation Supporting Documentation
- Appendix 6. Additional Data Requested by Client

## **DATA QUALITY OBJECTIVES**

### **Holding Times**

Analytical holding times were assessed to ascertain whether the holding time requirements were met by the laboratory. The holding time requirements are as follows: Samples must be extracted within 14 days of the date of sample collection and analyzed within 40 days from the date of extraction.

If holding times are exceeded, but not by greater than two times the limit, all associated sample results are qualified as estimates and flagged "J" for detects and "UJ" for non-detects. If holding times are exceeded by greater than two times the limit, all

associated detectable sample results are qualified as estimates and flagged "J" and all non-detects are rejected and flagged "UR".

All holding times were acceptable.

#### **Method Blanks**

Method blank analyses are conducted to determine the extent of laboratory contamination introduced through sampling, sample preparation and analysis. At least one acceptable method blank analysis must be conducted for every 20 samples. No contaminants should be present in the method blank. Analytical results for analytes present in any sample at less than five times the concentration of that analyte found in the associated blank are qualified as non-detects and flagged "U". Common laboratory contaminants present in samples at less than ten times the concentration of that analyte found in the associated blank are qualified as non-detects. If a sample result is less than the CRQL and is less than five times (or less than ten times for lab contaminants) the highest associated blank result, the sample result value is raised to the CRQL level and qualified as undetected "U".

All method blank results were acceptable.

#### Field Blanks

No field blank was submitted for analysis.

#### **Accuracy**

##### Matrix Spike/Matrix Spike Duplicate & Blank Spike Recoveries

Matrix spike/matrix spike duplicate analyses are used to assess the analytical accuracy of the reported data and the effect of the matrix on the ability to accurately quantify sample concentrations. Matrix spike/matrix spike duplicate analyses are performed in duplicate using five compounds for which percent recoveries must be within a range of 50-150% or within laboratory control limits. If spike recoveries are outside control limits, detected sample results less than five times the spike concentration are qualified as estimates and flagged "J". Undetected sample results with spike recoveries below control limits are qualified as estimates and flagged "UJ". Undetected sample results are not qualified if the spike recovery is above control limits. Sample results greater than five times the spike concentration require no qualification.

All accuracy results were acceptable.



### Surrogate Recovery

The analyses of surrogate compounds provide a measure of performance for individual samples. Matrix-specific surrogate compound recovery control windows have been established by the EPA CLP program. If two surrogates of the same class of compounds (base/neutral or acid) are out of control limits, all associated sample results greater than the contract required quantitation limit (CRQL) are qualified as estimates and flagged "J". Sample results less than the CRQL and below the lower control limit are qualified as estimates and flagged "UJ". Sample results less than the CRQL with recoveries above the upper control limit require no qualification. If a surrogate recovery is less than 10%, detects are qualified as estimates and flagged "J" and nondetects are rejected and flagged "UR".

All surrogate results were acceptable.

### • **Precision**

#### Matrix Spike/Matrix Spike Duplicate Samples

Matrix spike (MS)/matrix spike duplicate (MSD) results provide matrix-specific information on the precision of the method for specific target compound classes. Precision is expressed by the relative percent difference (RPD) between the recoveries of duplicate matrix spike analyses performed on a sample. Samples results must be within RPD limits of +/-30%. If RPD values are out of specification and the sample concentration is less than five times the spike concentration, all associated detected sample results are qualified as estimates and flagged "J". If RPD values are out of specification and the sample concentration is greater than five times the spike concentration, no qualification is required.

All duplicate results were acceptable.

#### Field Duplicate Samples

One set of field duplicates (J1V846/J1V848) were submitted for analysis. Laboratory duplicates are compared using the same criteria as for laboratory duplicates. All field duplicate results were acceptable.

### • **Analytical Detection Levels**

Reported analytical detection levels are compared against the required quantitation limits (RQL's) to ensure that laboratory detection levels meet the required criteria. All analytes met the RQL.

### **Completeness**

Data package No. JP1000 was submitted for validation and verified for completeness. Completeness is based on the percentage of data determined to be valid (i.e., not rejected). The completion percentage was 100%.

### **MAJOR DEFICIENCIES**

None found.

### **MINOR DEFICIENCIES**

None found.

### **REFERENCES**

Washington Closure Hanford Contract #S00W307A00 (March 2008), *Data Validation Services*, March 2008.

DOE/RL-96-22, Rev. 5, *100 Area Remedial Action Sampling and Analysis Plan*, U.S. Department of Energy, September 2009.

**Appendix 1**  
**Glossary of Data Reporting Qualifiers**

Qualifiers which may be applied by data validators in compliance with the WCH validation SOW are as follows:

- U - Indicates the compound or analyte was analyzed for and not detected in the sample. The value reported is the same quantitation limit corrected for sample dilution and moisture content by the laboratory.
- UJ - Indicates the compound or analyte was analyzed for and not detected in the sample. Due to a minor QC deficiency identified during the data validation, the associated quantitation limit is an estimate.
- J - Indicates the compound or analyte was analyzed for and detected. Due to a minor QC deficiency identified during the data validation, the associated quantitation limit is an estimate.
- R - Indicates the compound or analyte was analyzed for, detected, and due to an identified major QC deficiency, the data are unusable.
- UR - Indicates the compound or analyte was analyzed for and not detected in the sample. Additionally, the data is unusable due to an identified major QC deficiency.
- NJ - Indicates presumptive evidence of a compound at an estimated value. The data may not be valid for some specific applications (i.e., usable for decision-making purposes).
- N - Indicates presumptive evidence of a compound. The data may not be valid for some specific applications usable for decision-making purposes).

**Appendix 2**  
**Summary of Data Qualification**

# POLYAROMATIC HYDROCARBON DATA QUALIFICATION SUMMARY\*

<b>SDG: JP1000</b>	<b>REVIEWER: ELR</b>	<b>Project: 600-326</b>	<b>PAGE <u>1</u> OF <u>1</u></b>
<b>COMMENTS: No qualifiers assigned</b>			

\* - The Qualified Data Summary Table includes laboratory applied "U" qualifiers not specifically identified here. The laboratory applied "U" qualifiers are included to minimize misinterpretation of results contained in the table.

**Appendix 3**  
**Annotated Laboratory Reports**

# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Client Sample ID: J1V846

Lab Sample ID: 280-74906-1

Client Matrix: Solid

% Moisture: 9.7

Date Sampled: 10/01/2015 0755

Date Received: 10/02/2015 0935

## 8310 PAHs (HPLC)

Analysis Method: 8310	Analysis Batch: 280-298048	Instrument ID: CHHPLC_G
Prep Method: 3550C	Prep Batch: 280-297792	Initial Weight/Volume: 30.3 g
Dilution: 1.0		Final Weight/Volume: 4 mL
Analysis Date: 10/06/2015 1456		Injection Volume: 20 uL
Prep Date: 10/04/2015 0938		Result Type: PRIMARY

Analyte	DryWt Corrected: Y	Result (ug/Kg)	Qualifier	MDL	RL
Acenaphthene		11	U	11	110
Acenaphthylene		9.9	U	9.9	110
Anthracene		3.3	U	3.3	22
Benzo[a]anthracene		3.5	U	3.5	16
Benzo[a]pyrene		7.0	U	7.0	16
Benzo[b]fluoranthene		4.6	U	4.6	16
Benzo[g,h,i]perylene		7.9	U	7.9	33
Benzo[k]fluoranthene		4.3	U	4.3	16
Chrysene		5.3	U	5.3	44
Dibenzo(a,h)anthracene		12	U	12	33
Fluoranthene		14	U	14	44
Fluorene		5.8	U	5.8	33
Indeno[1,2,3-cd]pyrene		13	U	13	33
Naphthalene		13	U	13	110
Phenanthrene		13	U	13	44
Pyrene		13	U	13	44

Surrogate	%Rec	Qualifier	Acceptance Limits
Terphenyl-d14 (SUR)	91		72 - 115

*10/17/15*



# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Client Sample ID: J1V847

Lab Sample ID: 280-74906-2

Date Sampled: 10/01/2015 0720

Client Matrix: Solid

% Moisture: 11.5

Date Received: 10/02/2015 0935

## 8310 PAHs (HPLC)

Analysis Method: 8310

Analysis Batch: 280-298048

Instrument ID: CHHPLC\_G

Prep Method: 3550C

Prep Batch: 280-297792

Initial Weight/Volume: 32.3 g

Dilution: 1.0

Final Weight/Volume: 4 mL

Analysis Date: 10/06/2015 1527

Injection Volume: 20 uL

Prep Date: 10/04/2015 0938

Result Type: PRIMARY

Analyte	DryWt Corrected: Y	Result (ug/Kg)	Qualifier	MDL	RL
Acenaphthene		10	U	10	100
Acenaphthylene		9.4	U	9.4	100
Anthracene		3.2	U	3.2	21
Benzo[a]anthracene		4.0	J X	3.3	16
Benzo[a]pyrene		21		6.7	16
Benzo[b]fluoranthene		20		4.4	16
Benzo[g,h,i]perylene		7.6	U	7.6	31
Benzo[k]fluoranthene		4.1	U	4.1	16
Chrysene		5.1	U N	5.1	42
Dibenzo(a,h)anthracene		12	U	12	31
Fluoranthene		36	J	14	42
Fluorene		5.5	U	5.5	31
Indeno[1,2,3-cd]pyrene		16	J	13	31
Naphthalene		13	U	13	100
Phenanthrene		17	J X	13	42
Pyrene		13	U	13	42

Surrogate	%Rec	Qualifier	Acceptance Limits
Terphenyl-d14 (SUR)	94		72 - 115

10/17/15

# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Client Sample ID: J1V848

Lab Sample ID: 280-74906-3

Date Sampled: 10/01/2015 0755

Client Matrix: Solid

% Moisture: 8.5

Date Received: 10/02/2015 0935

## 8310 PAHs (HPLC)

Analysis Method: 8310

Analysis Batch: 280-298048

Instrument ID: CHHPLC\_G

Prep Method: 3550C

Prep Batch: 280-297792

Initial Weight/Volume: 31.8 g

Dilution: 1.0

Final Weight/Volume: 4 mL

Analysis Date: 10/06/2015 1658

Injection Volume: 20 uL

Prep Date: 10/04/2015 0938

Result Type: PRIMARY

Analyte	DryWt Corrected: Y	Result (ug/Kg)	Qualifier	MDL	RL
Acenaphthene		30	J	10	100
Acenaphthylene		9.3	U	9.3	100
Anthracene		3.1	U	3.1	21
Benzo[a]anthracene		3.3	U	3.3	15
Benzo[a]pyrene		6.6	U	6.6	15
Benzo[b]fluoranthene		4.3	U	4.3	15
Benzo[g,h,i]perylene		7.4	U	7.4	31
Benzo[k]fluoranthene		4.1	U	4.1	15
Chrysene		5.0	U	5.0	41
Dibenzo(a,h)anthracene		11	U	11	31
Fluoranthene		13	U	13	41
Fluorene		5.4	U	5.4	31
Indeno[1,2,3-cd]pyrene		12	U	12	31
Naphthalene		12	U	12	100
Phenanthrene		12	U	12	41
Pyrene		12	U	12	41

Surrogate	%Rec	Qualifier	Acceptance Limits
Terphenyl-d14 (SUR)	91		72 - 115

10/17/15

## **Appendix 4**

### **Laboratory Narrative and Chain-of-Custody Documentation**

## CASE NARRATIVE

Client: Washington Closure Hanford

Project: WASHINGTON CLOSURE HANFORD

Job Number: 280-74906-1

SDG #: JP1000

SAF#: RC-232

Date SDG Closed: October 2, 2015

Data Deliverable: 7 Day / Summary

<u>CLIENT ID</u>	<u>LAB ID</u>	<u>ANALYSES REQUESTED</u>	<u>ANALYSES PERFORMED</u>
J1V846	280-74906-1	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V847	280-74906-2	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V848	280-74906-3	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V849	280-74906-4	6010	6010B

I certify that this data package is in compliance with the SOW, both technically and for completeness, for other than the conditions detailed in this Case Narrative. Release of the data contained in this hard copy data package has been authorized by the Laboratory Manager or a designee, as verified by the signature on the Report Cover.

With exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. All laboratory quality control samples analyzed in conjunction with the samples in this project were within established control limits, with any exceptions noted. Calculations are performed before rounding to avoid round-off errors in calculated results.

This report includes reporting limits (RLs) less than TestAmerica Denver's practical quantitation limits. These reporting limits are being used specifically at the client's request to meet the needs of this project. Please note that data are not normally reported to these levels without qualification, since they are inherently less reliable and potentially less defensible than required by the current NELAC standards.

The results, RLs and MDLs included in this report have been adjusted for dry weight, as appropriate.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

### RECEIPT

The samples were received on 10/2/2015 9:35 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperature of the cooler at receipt was 3.3° C.

### GC SEMIVOLATILES - SW846 8081A - Pesticides

The organic prep laboratory noted that the samples presented in this report required a Florisil clean-up to reduce matrix interferences.

The laboratory noted that the samples presented in this report required a mercury clean-up to reduce matrix interferences caused by sulfur.

The RPD between the primary and confirmation columns exceeded 40% for 4,4'-DDE in sample J1V847. The higher of the two values has been reported. The result has been flagged with a "Y".

The MS/MSD performed on sample J1V846 exhibited spike compound recoveries outside the control limits, and the associated sample results have been flagged "N". In addition, the RPD limit was exceeded for 4,4'-DDD and 4,4'-DDE. The acceptable LCS analysis data indicated that the analytical system was operating within control; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

### HPLC - SW846 8310 - PAHs

The RPD between the primary and confirmation columns exceeded 40% for Benzo[a]anthracene and Phenanthrene in sample J1V847. The lower of the two values has been reported, as matrix interference is evident on both columns. The results have been flagged with an "X".

The MS/MSD performed on sample J1V847 exhibited percent recoveries outside the control limits for Chrysene, and the associated sample result has been flagged "N". The acceptable LCS analysis data indicated that the analytical system was operating within control; therefore, corrective action is deemed unnecessary.

Continuing Calibration Verification (CCV) standards associated with samples in analysis batch 280-298048 exhibited %Difference (%D) values >15%, biased high, for Benzo[g,h,i]perylene, Dibenzo[a,h]anthracene and Fluoranthene. The samples associated with these CCVs are either non-detect or less than the reporting limit; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

#### **TOTAL METALS - SW846 6010B**

Serial dilution of a digestate in batch 280-297784 indicates that physical and chemical interferences are present for Iron and Zinc. Results have been flagged with an "X".

Low levels of Barium, Calcium, Chromium, Magnesium and Nickel are present in the method blank associated with batch 280-297784. Because the concentrations in the method blank are not present at levels greater than half the reporting limit, corrective action is deemed unnecessary.

Zinc is present in the method blank associated with batch 280-297784 at 0.797 mg/kg, which is greater than half the project specific reporting limit (PSRL) of 1 mg/kg. TestAmerica's practical quantitation limit (PQL) for Zinc is 3 mg/kg. The laboratory cannot maintain system cleanliness at this low level; therefore, corrective action is not initiated. It can be noted that the concentration found in the method blank is less than half of the laboratory standard PQL, and with the exception of 'blank' sample J1V849, the associated sample amounts are greater than twenty times the method blank concentration.

Iron, a common laboratory contaminant, is present at a level greater than the reporting limit in the method blank associated with batch 280-297784. As the associated sample amounts are greater than twenty times the method blank concentration, corrective action is deemed unnecessary.

It can be noted that the sample amount was greater than four times the spike amount for Aluminum, Iron and Manganese in the Matrix Spike performed on sample J1V846; therefore, control limits are not applicable.

Silicon was recovered outside the control limits in the Matrix Spike performed on sample J1V846, and the associated sample result has been flagged "N". There is no indication that the analytical system was operating out of control, and method accuracy has been verified by the acceptable LCS analysis data; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

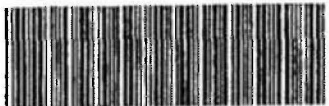
#### **GENERAL CHEMISTRY - SW846 9056M - ANIONS**

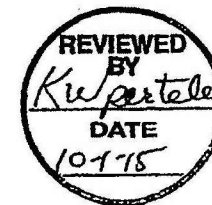
Each sample is analyzed to achieve the lowest possible reporting limits within the constraints of the method. Due to high constituent concentration, the Sulfate analysis of sample J1V847 had to be performed at a dilution, and the associated result has been flagged with a "D". The reporting limit has been adjusted relative to the dilution required.

Sulfate is present in the method blank associated with batch 280-297863 at 7.21 mg/kg, which is greater than the project specific reporting limit (PSRL) of 5 mg/kg. TestAmerica's practical quantitation limit (PQL) for Sulfate is 50 mg/kg. The laboratory cannot maintain system cleanliness at this low level; therefore, corrective action is not initiated. It can be noted that the concentration found in the method blank is less than half of the laboratory standard PQL.

The Matrix Spike performed on sample J1V846 exhibited the percent recovery outside the control limits for Fluoride, and the associated sample result has been flagged "N". There is no indication that the analytical system was operating out of control, and method accuracy has been verified by the acceptable LCS analysis data; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

Washington Closure Hanford				CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST				RC-232-102		Page 1 of 1		
Collector CRAIG, JC				Company Contact Joan Kessner		Telephone No. 375-4688		Project Coordinator KESSNER, JH		Price Code 83 B		
Project Designation 100-IU-2 & 100-IU-6 Remaining Waste Sites				Sampling Location 600-326 (Areas 1 and 2) verification		SAF No. RC-232		Data Turnaround 7 days				
Ice Chest No. AFS-04-008				Field Logbook No. EL-1667-03		COA 0603262000		Method of Shipment Fed Ex				
Shipped To TestAmerica Denver				Offsite Property No. A131443		Bill of Lading/Air Bill No. See ASPC						
Other Labs Shipped To N/A TRG 10-1-15				Preservation		Cool <=6C	Cool <=6C	Cool <=6C	Cool <=6C	 280-74906 Chain of Custody		
				Type of Container		G/P	aG	aG	G/P			
POSSIBLE SAMPLE HAZARDS/REMARKS None				No. of Container(s)		1	1	1	1			
				Volume		250mL	250mL	250mL	250mL			
Special Handling and/or Storage Cooling as required				Sample Analysis		See item (1) in Special Instructions	PAHs - 8310	Pesticides - 8081	See item (2) in Special Instructions			
Page				Sample No.	Matrix	Sample Date	Sample Time					
JP846				SOIL	10/01/15	0755	X	X	X	X		
JP847				SOIL	10/01/15	0720	X	X	X	X		
JP848				SOIL	10/01/15	0755	X	X	X	X		
JP849				SOIL	10/01/15	0743	X	X	X	X		
CHAIN OF POSSESSION				Sign/Print Names				SPECIAL INSTRUCTIONS (1) ICP Metals - 6010TR (Close-out List) (Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silicon, Silver, Sodium, Vanadium, Zinc) (2) IC Anions - 9058 Modified (Bromide, Chloride, Fluoride, Nitrogen in Nitrate, Nitrogen in Nitrite, Phosphorous in phosphate, Sulfate)  3.2 IKS+0.1 T. L. by M7 10-1-15				
Relinquished By/Removed From J. L. C. 10/1/15				Received By/Stored In J. L. C. 10/1/15								
Relinquished By/Removed From J. L. C. 10/1/15				Received By/Stored In J. L. C. 10/1/15								
Relinquished By/Removed From J. L. C. 10-1-15 1800				Received By/Stored In Fed Ex 10-1-15								
Relinquished By/Removed From				Received By/Stored In 10-2-15 9:35								
Relinquished By/Removed From				Received By/Stored In								
Relinquished By/Removed From				Received By/Stored In								
Relinquished By/Removed From				Received By/Stored In								
FINAL SAMPLE DISPOSITION				Disposed By				Date/Time				



JP1000

**Appendix 5**  
**Data Validation Supporting Documentation**

## GENERAL ORGANIC DATA VALIDATION CHECKLIST

VALIDATION LEVEL:	A	B	<b>C</b>	D	E
PROJECT:	600-320		DATA PACKAGE: JP1000		
VALIDATOR:	ELR	LAB: TAC	DATE: 10/15/15		
		SDG: JP1000			
ANALYSES PERFORMED					
8015	8021	8141	8151	8315	<b>8310</b>
		WTPH-HCID	WTPH-G	WTPH-D	
SAMPLES/MATRIX:					
J1V846 J1V847 J1V848					
Soil					

## 1. DATA PACKAGE COMPLETENESS AND CASE NARRATIVE

Technical verification documentation present? ..... Yes **No** N/A

Comments: \_\_\_\_\_

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## 2. INSTRUMENT TUNING AND CALIBRATION (Levels D and E)

Initial calibrations acceptable? ..... Yes No **N/A**Continuing calibrations acceptable? ..... Yes No **N/A**Standards traceable? ..... Yes No **N/A**Standards expired? ..... Yes No **N/A**Calculation check acceptable? ..... Yes No **N/A**

Comments: \_\_\_\_\_

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**GENERAL ORGANIC DATA VALIDATION CHECKLIST****3. BLANKS (Levels B, C, D, and E)**

Calibration blanks analyzed? (Levels D, E) ..... Yes No N/A

Calibration blank results acceptable? (Levels D, E) ..... Yes No N/A

Laboratory blanks analyzed? ..... Yes No N/A

Laboratory blank results acceptable? ..... Yes No N/A

Field/trip blanks analyzed? (Levels C, D, E) ..... Yes No N/A

Field/trip blank results acceptable? (Levels C, D, E) ..... Yes No N/A

Transcription/calculation errors? (Levels D, E) ..... Yes No N/A

Comments: no FB

**4. ACCURACY (Levels C, D, and E)**

Surrogates/system monitoring compounds analyzed? ..... Yes No N/A

Surrogate/system monitoring compound recoveries acceptable? ..... Yes No N/A

Surrogates traceable? (Levels D, E) ..... Yes No N/A

Surrogates expired? (Levels D, E) ..... Yes No N/A

MS/MSD samples analyzed? ..... Yes No N/A

MS/MSD results acceptable? ..... Yes No N/A

MS/MSD standards NIST traceable? (Levels D, E) ..... Yes No N/A

MS/MSD standards expired? (Levels D, E) ..... Yes No N/A

LCS/BSS samples analyzed? ..... Yes No N/A

LCS/BSS results acceptable? ..... Yes No N/A

Standards traceable? (Levels D, E) ..... Yes No N/A

Standards expired? (Levels D, E) ..... Yes No N/A

Transcription/calculation errors? (Levels D, E) ..... Yes No N/A

Performance audit sample(s) analyzed? ..... Yes No N/A

Performance audit sample results acceptable? ..... Yes No N/A

Comments: no PAH

**GENERAL ORGANIC DATA VALIDATION CHECKLIST****5. PRECISION (Levels C, D, and E)**

Duplicate RPD values acceptable? ..... ☒ Yes No N/A

Duplicate results acceptable? ..... ☒ Yes No N/A

MS/MSD standards NIST traceable? (Levels D, E) ..... Yes No ☒ N/A

MS/MSD standards expired? (Levels D, E) ..... ☒ Yes No ☒ N/A

Field duplicate RPD values acceptable? ..... ☒ Yes No N/A

Field split RPD values acceptable? ..... Yes No ☒ N/A

Transcription/calculation errors? (Levels D, E) ..... Yes No ☒ N/A

Comments: \_\_\_\_\_

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**6. HOLDING TIMES (all levels)**

Samples properly preserved? ..... ☒ Yes No N/A

Sample holding times acceptable? ..... ☒ Yes No N/A

Comments: \_\_\_\_\_

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**GENERAL ORGANIC DATA VALIDATION CHECKLIST****8. COMPOUND IDENTIFICATION, QUANTITATION, AND DETECTION LIMITS (all levels)**

Results reported for all requested analyses? ..... Yes No N/A  
Results supported in the raw data? (Levels D, E) ..... Yes No N/A  
Samples properly prepared? (Levels D, E) ..... Yes No N/A  
Detection limits meet RDL? ..... Yes No N/A  
Transcription/calculation errors? (Levels D, E) ..... Yes No N/A

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_**9. SAMPLE CLEANUP (Levels D and E)**

Fluoriscil ® (or other absorbant) cleanup performed? ..... Yes No N/A  
Lot check performed? ..... Yes No N/A  
Check recoveries acceptable? ..... Yes No N/A  
Check materials traceable? ..... Yes No N/A  
Check materials Expired? ..... Yes No N/A  
Analytical batch QC given similar cleanup? ..... Yes No N/A  
Transcription/Calculation Errors? ..... Yes No N/A

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Appendix 6**  
**Additional Documentation Requested by Client**

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Method Blank - Batch: 280-297792

Method: 8310

Preparation: 3550C

Lab Sample ID: MB 280-297792/1-A  
 Client Matrix: Solid  
 Dilution: 1.0  
 Analysis Date: 10/06/2015 1355  
 Prep Date: 10/04/2015 0938  
 Leach Date: N/A

Analysis Batch: 280-298048  
 Prep Batch: 280-297792  
 Leach Batch: N/A  
 Units: ug/Kg

Instrument ID: CHHPLC\_G  
 Lab File ID: G1006006.D  
 Initial Weight/Volume: 31.5 g  
 Final Weight/Volume: 4 mL  
 Injection Volume: 20 uL  
 Column ID: PRIMARY

Analyte	Result	Qual	MDL	RL
Acenaphthene	9.5	U	9.5	95
Acenaphthylene	8.6	U	8.6	95
Anthracene	2.9	U	2.9	19
Benzo[a]anthracene	3.0	U	3.0	14
Benzo[a]pyrene	6.1	U	6.1	14
Benzo[b]fluoranthene	4.0	U	4.0	14
Benzo[g,h,i]perylene	6.9	U	6.9	29
Benzo[k]fluoranthene	3.8	U	3.8	14
Chrysene	4.6	U	4.6	38
Dibenzo(a,h)anthracene	10	U	10	29
Fluoranthene	12	U	12	38
Fluorene	5.0	U	5.0	29
Indeno[1,2,3-cd]pyrene	11	U	11	29
Naphthalene	11	U	11	95
Phenanthrene	11	U	11	38
Pyrene	11	U	11	38

Surrogate	% Rec	Acceptance Limits
Terphenyl-d14 (SUR)	98	72 - 115

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

Lab Control Sample - Batch: 280-297792

Method: 8310

Preparation: 3550C

Lab Sample ID: LCS 280-297792/2-A  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/06/2015 1426  
Prep Date: 10/04/2015 0938  
Leach Date: N/A

Analysis Batch: 280-298048  
Prep Batch: 280-297792  
Leach Batch: N/A  
Units: ug/Kg

Instrument ID: CHHPLC\_G  
Lab File ID: G1006007.D  
Initial Weight/Volume: 30.7 g  
Final Weight/Volume: 4 mL  
Injection Volume: 20 uL  
Column ID: PRIMARY

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Acenaphthene	1950	1700	87	75 - 116	
Acenaphthylene	1950	1660	85	66 - 115	
Anthracene	1950	1620	83	71 - 115	
Benzo[a]anthracene	1950	1790	92	77 - 120	
Benzo[a]pyrene	1950	1840	94	69 - 115	
Benzo[b]fluoranthene	1950	1770	91	56 - 115	
Benzo[g,h,i]perylene	1950	1970	101	72 - 120	
Benzo[k]fluoranthene	1950	1830	94	76 - 115	
Chrysene	1950	1760	90	79 - 115	
Dibenzo(a,h)anthracene	1950	1780	91	72 - 115	
Fluoranthene	1950	1740	89	77 - 115	
Fluorene	1950	1740	89	77 - 115	
Indeno[1,2,3-cd]pyrene	1950	1700	87	78 - 115	
Naphthalene	1950	1710	87	68 - 120	
Phenanthrene	1950	1680	86	75 - 115	
Pyrene	1950	1820	93	72 - 115	
Surrogate		% Rec		Acceptance Limits	
Terphenyl-d14 (SUR)		94		72 - 115	

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

**Matrix Spike/  
Matrix Spike Duplicate Recovery Report - Batch: 280-297792**

**Method: 8310  
Preparation: 3550C**

MS Lab Sample ID: 280-74906-2  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/06/2015 1557  
Prep Date: 10/04/2015 0938  
Leach Date: N/A

Analysis Batch: 280-298048  
Prep Batch: 280-297792  
Leach Batch: N/A

Instrument ID: CHHPLC\_G  
Lab File ID: G1006010.D  
Initial Weight/Volume: 30.6 g  
Final Weight/Volume: 4 mL  
Injection Volume: 20 uL  
Column ID: PRIMARY

MSD Lab Sample ID: 280-74906-2  
Client Matrix: Solid  
Dilution: 1.0  
Analysis Date: 10/06/2015 1628  
Prep Date: 10/04/2015 0938  
Leach Date: N/A

Analysis Batch: 280-298048  
Prep Batch: 280-297792  
Leach Batch: N/A

Instrument ID: CHHPLC\_G  
Lab File ID: G1006011.D  
Initial Weight/Volume: 30.5 g  
Final Weight/Volume: 4 mL  
Injection Volume: 20 uL  
Column ID: PRIMARY

Analyte	% Rec.		Limit	RPD	RPD Limit	MS Qual	MSD Qual
	MS	MSD					
Acenaphthene	86	85	75 - 116	1	20		
Acenaphthylene	85	84	66 - 115	1	20		
Anthracene	89	86	71 - 115	3	20		
Benzo[a]anthracene	95	93	77 - 120	1	20		
Benzo[a]pyrene	86	85	69 - 115	1	20		
Benzo[b]fluoranthene	89	91	56 - 115	2	20		
Benzo[g,h,i]perylene	105	98	72 - 120	7	20		
Benzo[k]fluoranthene	100	99	76 - 115	1	20		
Chrysene	315	290	79 - 115	8	20	N	N
Dibenzo(a,h)anthracene	87	93	72 - 115	7	20		
Fluoranthene	91	90	77 - 115	1	20		
Fluorene	90	90	77 - 115	0	20		
Indeno[1,2,3-cd]pyrene	91	88	78 - 115	3	20		
Naphthalene	88	87	68 - 120	2	20		
Phenanthrene	79	81	75 - 115	2	20		
Pyrene	111	104	72 - 115	6	20		
Surrogate	MS % Rec		MSD % Rec	Acceptance Limits			
Terphenyl-d14 (SUR)	96		96	72 - 115			

Date: 19 August 2015  
To: Washington Closure Hanford Inc. (technical representative)  
From: ELR Consulting  
Project: 100-IU-2 & 100-IU-6 Remaining Waste Sites – Soil Full Protocol - Waste Site 600-326  
Subject: Wet Chemistry - Data Package No. JP1000-TAL

## **INTRODUCTION**

This memo presents the results of data validation on Data Package No. JP1000 prepared by TestAmerica Laboratories (TAL). A list of samples validated along with the analyses reported and the method of analysis is provided in the following table.

<b>Sample ID</b>	<b>Sample Date</b>	<b>Media</b>	<b>Validation</b>	<b>Analyte</b>
J1V846	10/1/15	Soil	C	See note 1
J1V847	10/1/15	Soil	C	See note 1
J1V848	10/1/15	Soil	C	See note 1

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Data validation was conducted in accordance with the Washington Closure Hanford (WCH) validation statement of work and the 100 Area Remedial Action Sampling and Analysis Plan (DOE/RL-96-22, September 2009). Appendices 1 through 6 provide the following information as indicated below:

- Appendix 1. Glossary of Data Reporting Qualifiers
- Appendix 2. Summary of Data Qualification
- Appendix 3. Annotated Laboratory Reports
- Appendix 4. Laboratory Narrative and Chain-of-Custody Documentation
- Appendix 5. Data Validation Supporting Documentation
- Appendix 6. Additional Documentation Requested by Client

## **DATA QUALITY PARAMETERS**

### **Holding Times**

Analytical holding times for metals are assessed to ascertain whether the holding time requirements were met by the laboratory. The holding time requirements are as follows: 28 days for chloride, fluoride, bromide, sulfate; and 48 hours for nitrate, nitrite and orthophosphate.

If holding times are exceeded, but not by greater than two times the limit, all associated sample results are qualified as estimates and flagged "J" for detects and "UJ" for non-detects. If holding times are exceeded by greater than two times the limit, all associated detectable sample results are qualified as estimates and flagged "J" and all non-detects are rejected and flagged "UR".



Due to the holding time being exceeded by greater than twice the limit, all detected nitrate, nitrate and orthophosphate results were qualified as estimates and flagged "J".

Due to the holding time being exceeded by greater than twice the limit, all undetected nitrate, nitrate and orthophosphate results were qualified as rejected and flagged "UR".

All other holding times were acceptable.

## • **Method Blanks**

### Method Blanks

Method blank analyses are performed to determine the extent of laboratory contamination introduced through sampling, sample preparation and analysis. At least one acceptable method blank analysis must be conducted for every 20 samples. No contaminants should be present in the method blank. All blank results must fall below the contract required detection limit (CRQL) to be acceptable.

All method blank results were acceptable.

### Field Blanks

No field blanks were submitted for analysis.

## • **Accuracy**

### Matrix Spike and Laboratory Control Sample

Matrix spike (MS) and laboratory control sample (LCS) analyses are used to assess the analytical accuracy of the reported data. The matrix spike is used to assess the effect of the matrix on the ability to accurately quantify sample concentrations. Recoveries must fall within the range of 70% to 130%. Samples with a recovery of less than 30% and a sample result below the IDL are rejected and flagged "UR". Samples with a recovery of 30% to 69% and a sample result less than the IDL are qualified "UJ". Samples with a recovery of greater than 130% or less than 70% and a sample result greater than the IDL are qualified as estimates and flagged "J". Finally, for samples with a recovery greater than 130% and a sample result less than the IDL, no qualification is required.

All accuracy results were acceptable.

## **Precision**

### **Laboratory Duplicate Samples**

Analytical precision is expressed by the relative percent differences (RPD) between the recoveries of matrix spike duplicate (MSD) analyses performed on a sample in the analytical batch. Precision may alternatively be assessed using unspiked duplicate analyses performed on a sample in the analytical batch. If both sample and replicate activities (concentrations) are greater than five times the CRDL and the RPD is less than 30%, no qualification is required. If either activity (concentration) is less than five times the CRDL, the RPD control limit is less than or equal to two times the CRDL. If the RPD is outside the applicable control limit, associated results are qualified as estimated detects or estimated non-detects.

All laboratory duplicate results were acceptable.

### **Field Duplicate**

One set of field duplicates (J1V846/J1V848) were submitted for analysis. Field duplicates are compared using the same criteria as for laboratory duplicates. All field duplicate results were acceptable.

## **Analytical Detection Levels**

Reported analytical detection levels are compared against the required quantitation limits (RQLs) to ensure that laboratory detection levels meet the required criteria. All analytes met the RQL.

## **Completeness**

Data package JP1000 was submitted for validation and verified for completeness. Completeness is based on the percentage of data determined to be valid (i.e., not rejected). The completion percentage was 71%.

## **MAJOR DEFICIENCIES**

The following major deficiencies were noted:

- Due to the holding time being exceeded by greater than twice the limit, all undetected nitrate, nitrate and orthophosphate results were qualified as rejected and flagged "UR".

Rejected data is unusable and should not be reported.

## **MINOR DEFICIENCIES**

The following minor deficiencies were noted:

- Due to the holding time being exceeded by greater than twice the limit, all detected nitrate, nitrate and orthophosphate results were qualified as estimates and flagged "J".

Data flagged "J" indicates that the associated concentration is an estimate, but under the WCH statement of work, the data may be usable for decision-making purposes. All other validated results are considered accurate within the standard error associated with the methods.

## **REFERENCES**

Washington Closure Hanford Contract #S00W307A00 (March 2008), *Data Validation Services*.

DOE/RL-96-22, Rev. 5, *100 Area Remedial Action Sampling and Analysis Plan*, U.S. Department of Energy, September 2009.

**Appendix 1**  
**Glossary of Data Reporting Qualifiers**

Qualifiers which may be applied by data validators in compliance with WCH validation SOW are as follows:

- U - Indicates the compound or analyte was analyzed for and not detected in the sample. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory.
- UJ - Indicates the compound or analyte was analyzed for and not detected in the sample. Due to a minor QC deficiency identified during the data validation, the associated quantitation limit is an estimate.
- J - Indicates the compound or analyte was analyzed for and detected. Due to a minor QC deficiency identified during the data validation, the associated concentration is an estimate, but the data are usable for decision-making purposes.
- BJ - Applied to inorganic analyses only. Indicates the analyte concentration was greater than the IDL but less than the CRDL and is considered an estimated value.
- R - Indicates the compound or analyte was analyzed for, detected, and due to an identified major QC deficiency, the data are unusable.
- UR - Indicates the compound or analyte was analyzed for and not detected in the sample. Additionally, the data is unusable due to an identified major QC deficiency.
- NJ - Indicates presumptive evidence of a compound at an estimated value. The data may not be valid for some specific applications (i.e., usable for decision-making purposes).
- N - Indicates presumptive evidence of a compound. The data may not be valid for some specific applications (i.e., usable for decision-making purposes).

**Appendix 2**  
**Summary of Data Qualification**

# WET CHEMISTRY DATA QUALIFICATION SUMMARY\*

<b>SDG: JP1000</b>	<b>REVIEWER: ELR</b>	<b>Project: 600-326</b>	<b>PAGE <u>1</u> OF <u>1</u></b>
<b>COMPOUND</b>	<b>QUALIFIER</b>	<b>SAMPLES AFFECTED</b>	<b>REASON</b>
Nitrate	J	All	Hold time
Nitrite Orthophosphate	UR	All	Hold time

\* - The Qualified Data Summary Table includes laboratory applied "U" qualifiers not specifically identified here. The laboratory applied "U" qualifiers are included to minimize misinterpretation of results contained in the table.

**Appendix 3**  
**Annotated Laboratory Reports**



# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

## General Chemistry

Client Sample ID: J1V846

Lab Sample ID: 280-74906-1

Date Sampled: 10/01/2015 0755

Client Matrix: Solid

% Moisture: 9.7

Date Received: 10/02/2015 0935

Analyte	Result	Qual	Units	MDL	RL	Dil	Method
Chloride-Soluble	2.2	U	mg/Kg	2.2	5.4	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1537				DryWt Corrected: Y
Nitrate as N-Soluble	0.71	B	mg/Kg	0.34	2.7	1.0	9056M
	Analysis Batch: 280-297862	Analysis Date: 10/05/2015	1537				DryWt Corrected: Y
Bromide-Soluble	0.42	U	mg/Kg	0.42	2.2	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1537				DryWt Corrected: Y
Nitrite as N-Soluble	0.36	U	mg/Kg	0.36	2.7	1.0	9056M
	Analysis Batch: 280-297862	Analysis Date: 10/05/2015	1537				DryWt Corrected: Y
Orthophosphate as P-Soluble	1.3	U	mg/Kg	1.3	5.4	1.0	9056M
	Analysis Batch: 280-297862	Analysis Date: 10/05/2015	1537				DryWt Corrected: Y
Sulfate-Soluble	43.4		mg/Kg	1.8	5.4	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1537				DryWt Corrected: Y
Fluoride-Soluble	0.89	U	mg/Kg	0.89	5.4	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1537				DryWt Corrected: Y
Analyte	Result	Qual	Units	RL	RL	Dil	Method
Percent Moisture	9.7		%	0.10	0.10	1.0	D-2216
	Analysis Batch: 280-297869	Analysis Date: 10/05/2015	1018				DryWt Corrected: N

*10/17/15*

# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1  
Sdg Number: JP1000

## General Chemistry

Client Sample ID: J1V847

Lab Sample ID: 280-74906-2  
Client Matrix: Solid

% Moisture: 11.5

Date Sampled: 10/01/2015 0720  
Date Received: 10/02/2015 0935

Analyte	Result	Qual	Units	MDL	RL	Dil	Method
Chloride-Soluble	2.9	B	mg/Kg	2.2	5.6	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1628				DryWt Corrected: Y
Nitrate as N-Soluble	2.8	J	mg/Kg	0.35	2.8	1.0	9056M
	Analysis Batch: 280-297862	Analysis Date: 10/05/2015	1628				DryWt Corrected: Y
Bromide-Soluble	0.44	U	mg/Kg	0.44	2.2	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1628				DryWt Corrected: Y
Nitrite as N-Soluble	0.38	U R	mg/Kg	0.38	2.8	1.0	9056M
	Analysis Batch: 280-297862	Analysis Date: 10/05/2015	1628				DryWt Corrected: Y
Orthophosphate as P-Soluble	1.4	U R	mg/Kg	1.4	5.6	1.0	9056M
	Analysis Batch: 280-297862	Analysis Date: 10/05/2015	1628				DryWt Corrected: Y
Sulfate-Soluble	3470	D	mg/Kg	9.5	27.9	5.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1707				DryWt Corrected: Y
Fluoride-Soluble	0.92	U	mg/Kg	0.92	5.6	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1628				DryWt Corrected: Y
Analyte	Result	Qual	Units	RL	RL	Dil	Method
Percent Moisture	11.5		%	0.10	0.10	1.0	D-2216
	Analysis Batch: 280-297869	Analysis Date: 10/05/2015	1018				DryWt Corrected: N

*h*  
10/17/15

# Analytical Data

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

## General Chemistry

Client Sample ID: J1V848

Lab Sample ID: 280-74906-3

Client Matrix: Solid

% Moisture: 8.5

Date Sampled: 10/01/2015 0755

Date Received: 10/02/2015 0935

Analyte	Result	Qual	Units	MDL	RL	Dil	Method
Chloride-Soluble	2.1	U	mg/Kg	2.1	5.3	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1645				DryWt Corrected: Y
Nitrate as N-Soluble	0.63	B J	mg/Kg	0.34	2.7	1.0	9056M
	Analysis Batch: 280-297862	Analysis Date: 10/05/2015	1645				DryWt Corrected: Y
Bromide-Soluble	0.42	U	mg/Kg	0.42	2.1	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1645				DryWt Corrected: Y
Nitrite as N-Soluble	0.36	U R	mg/Kg	0.36	2.7	1.0	9056M
	Analysis Batch: 280-297862	Analysis Date: 10/05/2015	1645				DryWt Corrected: Y
Orthophosphate as P-Soluble	1.3	U R	mg/Kg	1.3	5.3	1.0	9056M
	Analysis Batch: 280-297862	Analysis Date: 10/05/2015	1645				DryWt Corrected: Y
Sulfate-Soluble	43.4		mg/Kg	1.8	5.3	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1645				DryWt Corrected: Y
Fluoride-Soluble	0.88	U	mg/Kg	0.88	5.3	1.0	9056M
	Analysis Batch: 280-297863	Analysis Date: 10/05/2015	1645				DryWt Corrected: Y
Analyte	Result	Qual	Units	RL	RL	Dil	Method
Percent Moisture	8.5		%	0.10	0.10	1.0	D-2216
	Analysis Batch: 280-297869	Analysis Date: 10/05/2015	1018				DryWt Corrected: N

h  
10/17/15

## **Appendix 4**

### **Laboratory Narrative and Chain-of-Custody Documentation**

## CASE NARRATIVE

Client: Washington Closure Hanford

Project: WASHINGTON CLOSURE HANFORD

Job Number: 280-74906-1

SDG #: JP1000

SAF#: RC-232

Date SDG Closed: October 2, 2015

Data Deliverable: 7 Day / Summary

<u>CLIENT ID</u>	<u>LAB ID</u>	<u>ANALYSES REQUESTED</u>	<u>ANALYSES PERFORMED</u>
J1V846	280-74906-1	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V847	280-74906-2	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V848	280-74906-3	6010/9056M/8310/8081	6010B/9056M/8310/8081A
J1V849	280-74906-4	6010	6010B

I certify that this data package is in compliance with the SOW, both technically and for completeness, for other than the conditions detailed in this Case Narrative. Release of the data contained in this hard copy data package has been authorized by the Laboratory Manager or a designee, as verified by the signature on the Report Cover.

With exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. All laboratory quality control samples analyzed in conjunction with the samples in this project were within established control limits, with any exceptions noted. Calculations are performed before rounding to avoid round-off errors in calculated results.

This report includes reporting limits (RLs) less than TestAmerica Denver's practical quantitation limits. These reporting limits are being used specifically at the client's request to meet the needs of this project. Please note that data are not normally reported to these levels without qualification, since they are inherently less reliable and potentially less defensible than required by the current NELAC standards.

The results, RLs and MDLs included in this report have been adjusted for dry weight, as appropriate.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

### RECEIPT

The samples were received on 10/2/2015 9:35 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperature of the cooler at receipt was 3.3° C.

### GC SEMIVOLATILES - SW846 8081A - Pesticides

The organic prep laboratory noted that the samples presented in this report required a Florisil clean-up to reduce matrix interferences.

The laboratory noted that the samples presented in this report required a mercury clean-up to reduce matrix interferences caused by sulfur.

The RPD between the primary and confirmation columns exceeded 40% for 4,4'-DDE in sample J1V847. The higher of the two values has been reported. The result has been flagged with a "Y".

The MS/MSD performed on sample J1V846 exhibited spike compound recoveries outside the control limits, and the associated sample results have been flagged "N". In addition, the RPD limit was exceeded for 4,4'-DDD and 4,4'-DDE. The acceptable LCS analysis data indicated that the analytical system was operating within control; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

### HPLC - SW846 8310 - PAHs

The RPD between the primary and confirmation columns exceeded 40% for Benzo[a]anthracene and Phenanthrene in sample J1V847. The lower of the two values has been reported, as matrix interference is evident on both columns. The results have been flagged with an "X".

The MS/MSD performed on sample J1V847 exhibited percent recoveries outside the control limits for Chrysene, and the associated sample result has been flagged "N". The acceptable LCS analysis data indicated that the analytical system was operating within control; therefore, corrective action is deemed unnecessary.

Continuing Calibration Verification (CCV) standards associated with samples in analysis batch 280-298048 exhibited %Difference (%D) values >15%, biased high, for Benzo[g,h,i]perylene, Dibenzo[a,h]anthracene and Fluoranthene. The samples associated with these CCVs are either non-detect or less than the reporting limit; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

#### **TOTAL METALS - SW846 6010B**

Serial dilution of a digestate in batch 280-297784 indicates that physical and chemical interferences are present for Iron and Zinc. Results have been flagged with an "X".

Low levels of Barium, Calcium, Chromium, Magnesium and Nickel are present in the method blank associated with batch 280-297784. Because the concentrations in the method blank are not present at levels greater than half the reporting limit, corrective action is deemed unnecessary.

Zinc is present in the method blank associated with batch 280-297784 at 0.797 mg/kg, which is greater than half the project specific reporting limit (PSRL) of 1 mg/kg. TestAmerica's practical quantitation limit (PQL) for Zinc is 3 mg/kg. The laboratory cannot maintain system cleanliness at this low level; therefore, corrective action is not initiated. It can be noted that the concentration found in the method blank is less than half of the laboratory standard PQL, and with the exception of 'blank' sample J1V849, the associated sample amounts are greater than twenty times the method blank concentration.

Iron, a common laboratory contaminant, is present at a level greater than the reporting limit in the method blank associated with batch 280-297784. As the associated sample amounts are greater than twenty times the method blank concentration, corrective action is deemed unnecessary.

It can be noted that the sample amount was greater than four times the spike amount for Aluminum, Iron and Manganese in the Matrix Spike performed on sample J1V846; therefore, control limits are not applicable.

Silicon was recovered outside the control limits in the Matrix Spike performed on sample J1V846, and the associated sample result has been flagged "N". There is no indication that the analytical system was operating out of control, and method accuracy has been verified by the acceptable LCS analysis data; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.

#### **GENERAL CHEMISTRY - SW846 9056M - ANIONS**

Each sample is analyzed to achieve the lowest possible reporting limits within the constraints of the method. Due to high constituent concentration, the Sulfate analysis of sample J1V847 had to be performed at a dilution, and the associated result has been flagged with a "D". The reporting limit has been adjusted relative to the dilution required.

Sulfate is present in the method blank associated with batch 280-297863 at 7.21 mg/kg, which is greater than the project specific reporting limit (PSRL) of 5 mg/kg. TestAmerica's practical quantitation limit (PQL) for Sulfate is 50 mg/kg. The laboratory cannot maintain system cleanliness at this low level; therefore, corrective action is not initiated. It can be noted that the concentration found in the method blank is less than half of the laboratory standard PQL.

The Matrix Spike performed on sample J1V846 exhibited the percent recovery outside the control limits for Fluoride, and the associated sample result has been flagged "N". There is no indication that the analytical system was operating out of control, and method accuracy has been verified by the acceptable LCS analysis data; therefore, corrective action is deemed unnecessary.

No other anomalies were encountered.



**Appendix 5**  
**Data Validation Supporting Documentation**



## GENERAL CHEMISTRY ANALYSIS DATA VALIDATION CHECKLIST

VALIDATION LEVEL:	A	B	<u>C</u>	D	E
PROJECT: 600-32C			DATA PACKAGE: JP1000		
VALIDATOR: ELR		LAB: TAL		DATE: 10/15/15	
			SDG: JP1000		
ANALYSES PERFORMED					
Anions/IC	TOC	TOX	TPH-418.1	Oil and Grease	Alkalinity
Ammonia	BOD/COD	Chloride	Chromium-VI	pH	NO <sub>3</sub> /NO <sub>2</sub>
Sulfate	TDS	TKN	Phosphate		
SAMPLES/MATRIX					
J1V84C J1V847 J1V848					
5021					

## 1. DATA PACKAGE COMPLETENESS AND CASE NARRATIVE

Technical verification documentation present? ..... Yes No N/A

Comments: \_\_\_\_\_

## 2. INSTRUMENT PERFORMANCE AND CALIBRATIONS (Levels D and E)

Initial calibrations performed on all instruments? ..... Yes No N/AInitial calibrations acceptable? ..... Yes No N/AICV and CCV checks performed on all instruments? ..... Yes No N/AICV and CCV checks acceptable? ..... Yes No N/AStandards traceable? ..... Yes No N/AStandards expired? ..... Yes No N/ACalculation check acceptable? ..... Yes No N/A

Comments: \_\_\_\_\_

**GENERAL CHEMISTRY ANALYSIS DATA VALIDATION CHECKLIST****3. BLANKS (Levels B, C, D, and E)**

ICB and CCB checks performed for all applicable analyses? (Levels D, E)..... Yes No N/A

ICB and CCB results acceptable? (Levels D, E) ..... Yes No N/A

Laboratory blanks analyzed? ..... Yes No N/A

Laboratory blank results acceptable?..... Yes No N/A

Field blanks analyzed? (Levels C, D, E) ..... Yes No N/A

Field blank results acceptable? (Levels C, D, E) ..... Yes No N/A

Transcription/calculation errors? (Levels D, E)..... Yes No N/A

Comments: no FB**4. ACCURACY (Levels C, D, and E)**

Spike samples analyzed? ..... Yes No N/A

Spike recoveries acceptable? ..... Yes No N/A

Spike standards NIST traceable? (Levels D, E)..... Yes No N/A

Spike standards expired? (Levels D, E)..... Yes No N/A

LCS/BSS samples analyzed? ..... Yes No N/A

LCS/BSS results acceptable?..... Yes No N/A

Standards traceable? (Levels D, E)..... Yes No N/A

Standards expired? (Levels D, E) ..... Yes No N/A

Transcription/calculation errors? (Levels D, E)..... Yes No N/A

Performance audit sample(s) analyzed? ..... Yes No N/A

Performance audit sample results acceptable?..... Yes No N/A

Comments: no PA

**GENERAL CHEMISTRY ANALYSIS DATA VALIDATION CHECKLIST****5. PRECISION (Levels C, D, and E)**

Duplicate RPD values acceptable? ..... ☒ Yes No N/A

Duplicate results acceptable? ..... ☒ Yes No N/A

MS/MSD standards NIST traceable? (Levels D, E) ..... Yes No ☒ N/A

MS/MSD standards expired? (Levels D, E) ..... Yes No ☒ N/A

Field duplicate RPD values acceptable? ..... ☒ Yes No N/A

Field split RPD values acceptable? ..... Yes No ☒ N/A

Transcription/calculation errors? (Levels D, E) ..... Yes No ☒ N/A

Comments: \_\_\_\_\_

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**6. HOLDING TIMES (all levels)**

Samples properly preserved? ..... ☒ Yes No N/A

Sample holding times acceptable? ..... Yes ☒ No N/A

Comments: ortho, nitrate nitrite - 72x J/CR

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**GENERAL CHEMISTRY ANALYSIS DATA VALIDATION CHECKLIST**

**7. RESULT QUANTITATION AND DETECTION LIMITS (all levels)**

Results reported for all requested analyses? .....	<input checked="" type="radio"/> Yes	<input type="radio"/> No	<input type="radio"/> N/A
Results supported in the raw data? (Levels D, E) .....	<input type="radio"/> Yes	<input type="radio"/> No	<input checked="" type="radio"/> N/A
Samples properly prepared? (Levels D, E) .....	<input type="radio"/> Yes	<input type="radio"/> No	<input checked="" type="radio"/> N/A
Detection limits meet RDL? .....	<input checked="" type="radio"/> Yes	<input type="radio"/> No	<input type="radio"/> N/A
Transcription/calculation errors? (Levels D, E) .....	<input type="radio"/> Yes	<input type="radio"/> No	<input checked="" type="radio"/> N/A

Comments: \_\_\_\_\_

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**Appendix 6**  
**Additional Documentation Requested by Client**

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1  
Sdg Number: JP1000

### Method Blank - Batch: 280-297862

**Method: 9056M**  
**Preparation: N/A**

Lab Sample ID: MB 280-297896/2-A	Analysis Batch: 280-297862	Instrument ID: WC_IonChrom8
Client Matrix: Solid	Prep Batch: N/A	Lab File ID: 15.0000.d
Dilution: 1.0	Leach Batch: 280-297896	Initial Weight/Volume: 5 mL
Analysis Date: 10/05/2015 1724	Units: mg/Kg	Final Weight/Volume: 5 mL
Prep Date: N/A		
Leach Date: 10/05/2015 1321		

Analyte	Result	Qual	MDL	RL
Nitrate as N-Soluble	0.31	U	0.31	2.5
Nitrite as N-Soluble	0.34	U	0.34	2.5
Orthophosphate as P-Soluble	1.2	U	1.2	5.0

### Method Reporting Limit Check - Batch: 280-297862

**Method: 9056M**  
**Preparation: N/A**

Lab Sample ID: MRL 280-297862/3	Analysis Batch: 280-297862	Instrument ID: WC_IonChrom8
Client Matrix: Water	Prep Batch: N/A	Lab File ID: 03.0000.d
Dilution: 1.0	Leach Batch: N/A	Initial Weight/Volume: 5 mL
Analysis Date: 10/05/2015 1254	Units: mg/L	Final Weight/Volume: 5 mL
Prep Date: N/A		
Leach Date: N/A		

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Nitrate as N-Soluble	0.200	0.193	96	50 - 150	B
Nitrite as N-Soluble	0.200	0.171	85	50 - 150	B
Orthophosphate as P-Soluble	0.200	0.19	63	50 - 150	U

### Lab Control Sample - Batch: 280-297862

**Method: 9056M**  
**Preparation: N/A**

Lab Sample ID: LCS 280-297896/1-A	Analysis Batch: 280-297862	Instrument ID: WC_IonChrom8
Client Matrix: Solid	Prep Batch: N/A	Lab File ID: 07.0000.d
Dilution: 1.0	Leach Batch: 280-297896	Initial Weight/Volume: 5 mL
Analysis Date: 10/05/2015 1504	Units: mg/Kg	Final Weight/Volume: 5 mL
Prep Date: N/A		
Leach Date: 10/05/2015 1321		

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Nitrate as N-Soluble	50.0	49.94	100	90 - 110	
Nitrite as N-Soluble	50.0	49.86	100	90 - 110	
Orthophosphate as P-Soluble	50.0	50.10	100	90 - 110	

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

### Matrix Spike - Batch: 280-297862

Method: 9056M

Preparation: N/A

Lab Sample ID:	280-74906-1	Analysis Batch:	280-297862	Instrument ID:	WC_IonChrom8
Client Matrix:	Solid	Prep Batch:	N/A	Lab File ID:	11.0000.d
Dilution:	1.0	Leach Batch:	280-297896	Initial Weight/Volume:	5 mL
Analysis Date:	10/05/2015 1611	Units:	mg/Kg	Final Weight/Volume:	5 mL
Prep Date:	N/A				25 uL
Leach Date:	10/05/2015 1321				

Analyte	Sample Result/Qual	Spike Amount	Result	% Rec.	Limit	Qual
Nitrate as N-Soluble	0.71 B	54.4	56.97	103	80 - 120	
Nitrite as N-Soluble	0.36 U	54.4	55.07	101	80 - 120	
Orthophosphate as P-Soluble	1.3 U	54.4	49.43	91	80 - 120	

### Duplicate - Batch: 280-297862

Method: 9056M

Preparation: N/A

Lab Sample ID:	280-74906-1	Analysis Batch:	280-297862	Instrument ID:	WC_IonChrom8
Client Matrix:	Solid	Prep Batch:	N/A	Lab File ID:	10.0000.d
Dilution:	1.0	Leach Batch:	280-297896	Initial Weight/Volume:	5 mL
Analysis Date:	10/05/2015 1554	Units:	mg/Kg	Final Weight/Volume:	5 mL
Prep Date:	N/A				25 uL
Leach Date:	10/05/2015 1321				

Analyte	Sample Result/Qual	Result	RPD	Limit	Qual
Nitrate as N-Soluble	0.71 B	0.718	0.4	15	B
Nitrite as N-Soluble	0.36 U	0.37	NC	15	U
Orthophosphate as P-Soluble	1.3 U	1.4	NC	15	U

## Quality Control Results

Client: Washington Closure Hanford

Job Number: 280-74906-1

Sdg Number: JP1000

### Method Blank - Batch: 280-297863

Method: 9056M  
Preparation: N/A

Lab Sample ID:	MB 280-297896/2-A	Analysis Batch:	280-297863	Instrument ID:	WC_IonChrom8
Client Matrix:	Solid	Prep Batch:	N/A	Lab File ID:	15.0000.d
Dilution:	1.0	Leach Batch:	280-297896	Initial Weight/Volume:	5 mL
Analysis Date:	10/05/2015 1724	Units:	mg/Kg	Final Weight/Volume:	5 mL
Prep Date:	N/A				
Leach Date:	10/05/2015 1321				

Analyte	Result	Qual	MDL	RL
Chloride-Soluble	2.0	U	2.0	5.0
Bromide-Soluble	0.39	U	0.39	2.0
Sulfate-Soluble	7.21		1.7	5.0
Fluoride-Soluble	0.82	U	0.82	5.0

### Method Reporting Limit Check - Batch: 280-297863

Method: 9056M  
Preparation: N/A

Lab Sample ID:	MRL 280-297863/3	Analysis Batch:	280-297863	Instrument ID:	WC_IonChrom8
Client Matrix:	Water	Prep Batch:	N/A	Lab File ID:	03.0000.d
Dilution:	1.0	Leach Batch:	N/A	Initial Weight/Volume:	5 mL
Analysis Date:	10/05/2015 1254	Units:	mg/L	Final Weight/Volume:	5 mL
Prep Date:	N/A				
Leach Date:	N/A				

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Chloride-Soluble	2.50	2.49	99	50 - 150	B
Bromide-Soluble	0.200	0.212	106	50 - 150	
Sulfate-Soluble	2.50	2.49	99	50 - 150	B
Fluoride-Soluble	0.200	0.175	87	50 - 150	B

### Lab Control Sample - Batch: 280-297863

Method: 9056M  
Preparation: N/A

Lab Sample ID:	LCS 280-297896/1-A	Analysis Batch:	280-297863	Instrument ID:	WC_IonChrom8
Client Matrix:	Solid	Prep Batch:	N/A	Lab File ID:	07.0000.d
Dilution:	1.0	Leach Batch:	280-297896	Initial Weight/Volume:	5 mL
Analysis Date:	10/05/2015 1504	Units:	mg/Kg	Final Weight/Volume:	5 mL
Prep Date:	N/A				
Leach Date:	10/05/2015 1321				

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Chloride-Soluble	1000	1035	104	90 - 110	
Bromide-Soluble	50.0	48.67	97	90 - 110	
Sulfate-Soluble	1000	1006	101	90 - 110	
Fluoride-Soluble	50.0	49.58	99	90 - 110	